Capillary Suction Time (CST) Test:

Developments in testing methodology and reliability of results

Ola Sawalha

A thesis submitted for the degree of doctor of philosophy

The University of Edinburgh

(2010)

Declaration

I hereby declare that all the work in this thesis is my own and no part of this thesis has been used or submitted for any degree or professional qualification.

The knowledge and the help from the others during the course of this study have been properly referenced and acknowledged.

Ola Sawalha

2010

Acknowledgements

I would like to thank and appreciate the help and the assistance that I have received from my inspiring supervisor Prof. Miklas Scholz during my PhD research. Thanks also to my second supervisor, Dr. Vasileios Koutsos for his assistance particularly during the early stages of this study. I would like to show my gratitude to Prof. of Materials Chris Hall for his highly valuable scientific and mathematical inputs and for reviewing some parts of the thesis.

Special thanks goes to my sponsor: Triton Electronics Ltd. and Mr. Julian Tapp for partially funding this research and providing the required materials. Thanks also to the University of Edinburgh for providing most of the needed finical support for this research. I am also very thankful to the University of Edinburgh small project grants for their award toward this research.

Earnest gratefulness to Drs. Rut Besseling and Pierre Ballesta (School of Physics and Astronomy) for assisting me in using their rheology laboratory. Thanks also to Ms. Alanna Campbell for her assistance in particle size distributions measurements. I also appreciate the help that I have received from Mr. Jim Hutcheson, Mr. Douglas Carmichael, Mr. Derek Jardine, Mrs. Margaret Taylor, Mrs. Joan Birse, Mrs. Tanya Peshkur, and Dr. Peter Anderson for technical and clerical support.

Thanks also to those undergraduate and visiting students for their help in running some of the experiments of this research. Special thanks goes to Mr. Graeme Noble, Mr. Mark McMillan, Mr. Louis Marant, Mr. Theo Malzieu, Ms. Cecile Savary, and Ms. Dorthe Borges.

I am very thankful to my colleagues Dr. Paul Eke, Dr. Sara Kazemi-Yazdi, Dr. Piotr Grabowiecki, Dr. Atif Mustafa, Dr. Kiran Tota-Maharaj, Mrs. Qinili Yang, and Mr. Yu Dong for their great support, assistance, and friendship.

I would like to express my gratitude for my beloved parents, brothers and my sister for their unconditional love, unlimited supports, and encouragements.

The most special and big thanks goes to my husband Dr. Rami Sawalha for his great support, understanding, big assistance and encouragements all the way through the course of my PhD programme. The person that I have always found when I needed anything. Finally, special love goes to my sweet daughters Razan and Tala. They were the lights in my life when everything was dark and they were very patients and understanding to have a mom doing PhD.

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Abstract

The dewatering of wastewater sludge (slurry) is a routine operation at wastewater treatment plants, and the results of dewaterability tests underpin the selection of dewatering processes. The two most commonly applied dewaterability tests for this purpose are the capillary suction time (CST) test and the specific resistance to filtration (SRF) test. The aim of this research was to develop improved methods of estimating sludge dewaterability by modifying the components and procedures used in the standard CST test, and by exploring the causes of the high variability that confounds the interpretation of the CST test results. The applications of this research were to recommend alternative methodologies that would help to improve the accuracy and precision of the standard CST test device and procedures, and ideally reduce operational and consumable costs.

Multi-factorial experiments were designed to test the capillary suction times and the specific resistances to filtration of natural sludges and also of a synthetic medium which was formulated to simulate the properties of natural sludges. The applicability of altering the funnel geometry of the CST device, and the use of several alternative types of filter paper was evaluated. The applicability of incorporating stirring activity to eliminate or a least reduce sedimentation, and of adding a sealant at the bottom of the funnel, to eliminate or at least minimize unwanted filtrate leakage between the edge of the funnel and the filter paper, were studied. Experiments were performed to analyze the effects of temperature on the properties of sludges and the results of CST tests.

Improved methods of estimating sludge dewaterability were developed by modifying the components and procedures used in the standard CST test, and by

exploring the causes of variability in the test results. Stable synthetic sludges were successfully formulated to simulate the properties of natural sludges for experimental purposes. A rectangular funnel significantly reduced the variability and the time taken to conduct the CST test, relative to a circular funnel, particularly when testing heavy sludges. Whatman 17 chr (the most commonly used anisotropic filter paper) did not produce the most consistent CST test results in the shortest time. It is recommended that isotropic filter papers could be used, to lower the cost, reduce the test time, and improve the test precision. The addition of a sealant to the CST test apparatus also reduced the variability in the test results. No significant effects were found when a stirrer was added to the apparatus. The best line of fit to estimate filterability was defined by $\log_e Y = \beta_0 + \beta_1 \sqrt{x}$ where Y = the mean CST value (s); β_0 = the intercept (the predicted mean CST (s) when the distance[4] between electrodes of the CST device is zero); β_1 = the filterability (s/m^2); X = the distance[4] (m) between the electrodes of the CST device. Non-linear relationships were found between the CST test times and the temperature, associated with a complexity of effects of the temperature on sludge viscosity, filterability, settleability, desorptivity, and flocculation behaviour. It is recommended that the temperature should be recorded and controlled during the conduction of CST tests. SRF test results were predicted from the results of CST tests by the empirical model $\log_e SRF = 46.128 - 1.346\,T + 0.035\,T^2 + 13.760\,F/TSS$ where SRF is the specific resistance to filtration (m/kg); T is the temperature (ºC); F is the filterability ($\log_e s/m^2$) and TSS is the total suspended solids concentration (g/l).

Publications

Journal papers

Sawalha, O., and Scholz, M. 2007. Assessment of capillary suction time (CST) test methodologies. *Environmental Technology,* 28, 1377-1386.

Sawalha, O., and Scholz, M. 2009. Innovative enhancement of the design and precision of the capillary suction time testing device. *Water Environment Research,* 81, 2344-2352.

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Conference papers

Scholz M., Sawalha O. and Borges D. (2006), Review of capillary suction time testing. In Ubertini L. (ed.), *Proceedings of the Second International Association of Science and Technology for Development (IASTED) International Conference on 'Advanced Technology in the Environmental Field' (06-08/02/06), Lanzarote, Spain, ACTA Press, Calgary*. ISBN: 088986-552-3, 106-111.

Sawalha O. and Scholz M. (2006), Capillary suction time (CST): Evaluation of testing methodology and improvement of the apparatus. *4th National Conference held by The Chartered Institution of Water and Environmental Management (CIWEM) and Aqua Enviro Technology Transfer in Newcastle (12-14/09/06), Aqua Enviro Technology Transfer, Leeds, UK*. 7 pages on CD, ISBN: 1-903958-18-0.

Sawalha O., Scholz M. and Eke E. P. (2007), Improving the accuracy of capillary suction time testing. Kungolos A., Aravossis K., Karagiannidis A. and Samaras P. (Eds.). *Proceedings of the Society for Ecotoxicology Conference and the International Conference on Environmental Management, Engineering, Planning and Economics (24-28/06/2007), Skiathos Island, Greece. Grafima Publishing, Thessaloniki, Greece.* ISBN: 978-960-89818-0-5, Volume 2, 1431-1436.

Sawalha O. (2010), Effect of temperature on waste water filterability. *Graduate School of Engineering's Annual Postgraduate Research Conference (3/05/2010). Pentland Suite, John McIntyre Centre, Pollock Halls, University of Edinburgh, Uk.*

Chapter One

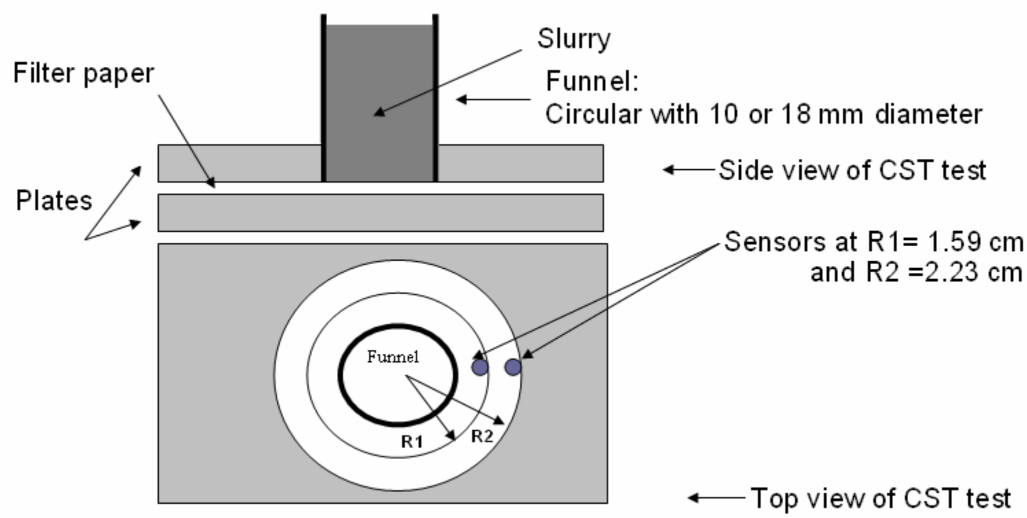
General Introduction

1.1 Background

The dewatering of sludge is defined as "the removal of enough of the liquid portion of the sludge so that it behaves as a solid" (Yukseler *et al.,* 2007). The dewatering of wastewater sludge (slurry) using evaporation beds, vacuum filters, belt and filter presses, centrifuges and other industrial drying processes is a routine operation at wastewater treatment plants (Scholz, 2005; Yukseler *et al*., 2007). The main reason for dewatering sludge is to reduce its volume by eliminating water, which lowers transportation costs, and facilitates storage. Dewatering also helps to stabilize sludge. In addition, sludge structure is improved by dewatering, so that it can be made into a fertilizer or soil conditioner which can be spread by agricultural equipment (Wang *et al.,* 2007). The results of dewaterability tests underpin the selection of dewatering processes, including the use of conditioners (Scholz, 2005; Yukseler, *et al.,* 2007). The two most commonly applied tests for this purpose are the capillary suction time (CST) test and the specific resistance to filtration (SRF) test.

The standard CST test was first developed by Gale and Baskerville (1967). The components include an open stainless steel cylindrical column or funnel with a Whatman 17 chr filter paper at the base, multiple electrodes, which serve to sense the movement of filtrate across the paper, and a timer. A diagram of capillary suction time test apparatus is shown in Figure 1.1.1. A sample of sludge is poured into the column, and the filtrate is extracted by capillary suction through the paper, so that a cake is formed on the filter. The distance the filtrate travels along the paper, as a function of time, is taken as a measure of the cake resistance, whilst the filter resistance is assumed to be negligible. The CST is the time taken for the filtrate to travel between two concentric and adjacent circles on the filter paper. If there is sufficient sludge to generate a force by capillary suction, then the test is relatively independent of the volume of the sample.

Figure 1.1.1 Diagram of capillary suction time test apparatus



The CST test is one of the most commonly used tests to determine sludge dewaterability (Lee and Hsu, 1994a). The CST test is widely used because it is quick, reliable, simple and inexpensive. It does not require an external source of pressure or suction, and the automated CST test device is easy to use and portable, so that tests can be performed in any location by persons with little training. However it suffers from several drawbacks. CST values are relatively specific to the sludge being tested, so that CST test data using different types of sludge are not necessarily comparable. In addition, the validity of the CST test has been questioned because replicated tests may yield variable results. It has been suggested that more research needs to focus on the factors associated with the precision of the results of CST tests (Scholz, 2005; 2006) providing a positive direction and rationale for the current study.

The SRF test is also widely used to estimate sludge dewaterability. The standard test uses a Buchner funnel apparatus, with a vacuum port and paper filter. A homogeneous sludge sample is poured into the funnel, the vacuum suction is applied, and the filtrate volume is measured at fixed time intervals, until the drainage stops. Theoretically, the plot of t/V versus V, where t = a given time and V = cumulative volume filtered, should be a straight line. The slope of this line is equivalent to the average SRF which is taken as a measure of the filterability or dewaterability of the sludge. If the slope is steep, the dewaterability is poor. The flatter the slope, the better is the dewaterability. The SRF test, however, is more difficult to execute, time-consuming, and expensive than the CST test, and no specific device to measure SRF is available (Ayol and Dentel, 2005; Teoh *et al.*, 2006; Yukseler *et. al*, 2007). Furthermore, differences in the apparatus and procedures used, e.g., the filter medium and the vacuum applied, have been found to cause variability in the results of SRF tests reported by different workers (Smollen, 1986a; 1986b).

The results of CST and SRF tests on the same sludge are correlated. Baskerville and Gale (1968) found that that sludge samples with high CST values also had high SRF values. Sludge filterability, which is a known function of SRF, was shown using mathematical Modelling to be estimable from CST values (Meeten and Smeulders, 1995). This model can be used to compute the sludge filterability, estimated as the slope of the regression line of successively recorded CST values versus the successive distances to the power four between the multiple electrodes of the CST device.

1.2 Aims and objectives

This study aimed to contribute towards the development of improved methods of estimating sludge dewaterability, by evaluating the effects of modifying specified components and procedures used in the standard CST test, and by exploring some of the causes of variability in the results of CST tests. It also aimed to relate the results of SRF tests to the results of CST tests. The focus of this investigation was essentially methodological; however, by consequence it was also highly statistical, due to the large number of quantitative variables and interacting factors involved in the experiments. The main objectives of this study were:

- To formulate a synthetic sludge to simulate the properties of natural sludge for experimental purposes;
- To evaluate the applicability of altering the funnel geometries (e.g., circular or rectangular; smaller or larger);
- To evaluate the applicability of several alternative filter papers (e.g., less expensive; isotropic; smaller pore diameter);

- To evaluate the applicability of incorporating stirring activity in the test device;
- To evaluate the applicability of adding a sealant at the bottom of the funnel, to eliminate or at least minimize unwanted filtrate leakage between the edge of the funnel and the filter paper, and potentially improve the test repeatability;
- To determine the effects of temperature on the results of CST tests;
- To model the effects of different sludge concentrations, and temperatures on the results of CST tests;
- To model the relationships between the results of SRF tests and CST tests;
- To recommend alternative methodologies that may help to improve the precision of the standard CST test device and procedures, and possibly reduce operational and consumable costs.

1.3 Literature Review

1.3.1 The practical applications of CST and SRF tests

CST test has a few engineering applications outside the wastewater treatment industry, such as the use of CST as a screening tool for predicting the relative rates of invasion of drilling fluids in permeable cores (Hoff and Growcock, 2004). The predominant application of CST and SRF tests, however, is to support industrial wastewater treatment processes. This is the application of interest to the current research. Since poor dewaterability may cause bottlenecks in the sludge handling sequence at treatment plants, CST and SRF tests are applied routinely as support operations, to determine the capacity of sludge to be dewatered, and to select the most appropriate mechanical drying processes (Scholz, 2005; Yukseler, *et al.,* 2007). CST and SRF tests are also applied to support the choice of a conditioning process to improve sludge dewaterability (Zhao, *et al.,* 2002). Conditioning is a pre-treatment, applied before mechanical dewatering, to enhance the dewaterability of the sludge. Sludge conditioning is achieved mainly through charge neutralization and polymer bridging. The optimum polymer dose is the amount used when the sludge particle charge is neutralized (Higgins *et al*., 2006). The main aim of conditioning is to promote the separation of flocs from the fluid to achieve a high solid content. Sludge dewaterability is strongly influenced by cations, specifically Potassium, Aluminium, Calcium, and ferric ions. Consequently, many inorganic substances, such as alum, ferric chloride, ferric sulphate, and lime are used in practice as sludge conditioners (Higgins and Novak, 1997; Novak and Park, 2004; Park *et al*., 2006; Nguyen *et al*., 2008). Organic additives for conditioning wastewater sludge have also been developed including cationic, anionic, and non-ionic polymers (Dentel, 1993; 2001).

Several researchers have compared the results of CST and SRF tests with respect to the use of sludge conditioners. Estimates of the CST and the SRF of conditioned sludge indicated similar dewaterability properties when inorganic conditioners were added to the sludge (Christensen *et al.,* 1993). When using organic polymer conditioners, however, the results of CST and SRF tests have been found to be inconsistent. Christensen *et al.,* (1993) reported that the optimal dose of organic conditioner determined using the SRF test was higher than the dose determined by use of the CST test. In contrast, Wu *et al.* (1997) discovered the opposite relationship. The optimal dose of organic polymer determined by use of the CST test was higher than that determined by use of SRF test.

Pan *et al.,* (2003) computed correlation coefficients to evaluate the strengths of the relationships between the results of CST and SRF tests and sludge dewaterability achieved by various conditioning and mechanical drying processes. CST tests did not reliably reflect the dewaterability of sludge by filter press drying. This was because pressure is exerted on the sludge during filter press drying, but no pressure is assumed when estimating dewaterability using CST tests. The SRF test was a better option to predict filter press dewatering because of the similarities in the filtration processes involved. Dewaterability by centrifuging was best reflected by the results of CST tests, because in both processes, no pressure is exerted on the sludge. When sludge was pre-treated at a freezing temperature, its dewaterability was generally unpredictable using CST and SRF tests. The low correlations between the results of the dewaterability tests were assumed to be associated with the changes in the physical structure of the sludge associated with freezing and thawing. Tao *et al* (2005) showed that the dewatering efficiency is negatively associated with freezing speed (lower dewatering efficiency at faster freezing speeds).

1.3.2. The formulation and use of synthetic sludges

The results of dewatering tests performed under different experimental conditions may be difficult to compare directly. This is because the properties of natural sludge samples are very variable, depending upon treatment plant operating conditions, and are highly dynamic, changing over time during transport, handling, and storage. A biological response starts when a sample of activated sludge from a treatment plant is kept aerated in the laboratory, which changes its chemical, biological, and physical properties (Sanin and Vesilind, 1996). Preservation of activated sludge in the laboratory, e.g. by freezing, also changes its properties (Pan *et* *al.,* 2003; Baudez *et al*., 2007). The unstable and unpredictable properties of natural organic sludges, even when sampled from the same source, imply that the results of CST and SRF tests, and other tests of sludge properties, such as settleability, may be difficult to reproduce experimentally. This problem has promoted the development and use of synthetic sludges. Numerous studies have demonstrated that synthetic sludges may be used as surrogate sludges for experimental purposes, particularly when the reproducibility of the test results is an important factor (Nguyen *et al.,* 2008).

It is essential that the dewatering and flocculation properties of synthetic sludges are similar to those of natural sludges, so that the results of experimental tests performed on the two types of sludge are comparable. Flocculation is a process that occurs when clays, organic polymers, microbial cells, and other small suspended charged particles become loosely attached together, and coagulate to form fragile solid structure known as flocs (Higgins *et al*., 2006). Flocculation in natural activated sludge is associated with the release of extracellular polymers, including proteins, polysaccharides and lipids, due to the metabolic activities of microorganisms, specifically bacteria, algae, protozoa, and fungi (Dignac *et al.,* 1988; Chundakkadu and Loosdrecht,1999). The flocs may float up to the top or settle down to the bottom, and they can be easily filtered, thereby influencing the results of CST and SRF tests (Ives, 1978).

Sanin and Vesilind (1996) were the first to formulate a synthetic sludge for experimental purposes, and they used it to postulate a mechanism to explain floc formation. Polystyrene latex particles were used to simulate bacteria. Alginate, a polysaccharide, was used to simulate extracellular polymers. The typical Calcium and extracellular polymer concentrations in activated sludge were replicated. Synthetic flocs were formed immediately following the addition of Calcium ions, and were similar in structure to the natural flocs in activated sludge. The role of Calcium is to provide bridging or binding cations, since they remove the repulsive barrier between negatively charged particles, and they have a high affinity for polymer, a process which is influenced by the pH of the medium (Gregor *et al.,* 1996). The synthetic sludge formulated by Sanin and Vesilind (1996) had similar dewatering properties to natural organic sludge. Using the SRF test, the filterability of the synthetic sludge was 4.32×10^{12} m/kg, which was within the range of 1.79×10^{12} to 6×10^{13} m/kg observed in natural waste activated sludge. The effects of a polyelectrolyte conditioner on the specific resistance to filtration of natural activated sludge and the synthetic organic sludge were found to be similar. Nguyen *et al.* (2008) similarly compared the effects of cationic polymers on the properties of synthetic and activated sludge. The two types of sludge had similar dewatering characteristics, measured using CST tests, after polymer conditioning. It was concluded that, although the synthetic sludge was not comparable to natural sludge in terms of its biological components, the synthetic sludge was a more stable material for experimental purposes in the laboratory since unlike natural sludge its physical and chemical structure was well defined and it remained stable over time.

Örmeci and Vesilind (2000) used the same synthetic sludge, based on polystyrene latex particles, alginate, and Calcium, with the addition of cellulose fibres to simulate the filamentous bacteria found in natural sludge. It was found that the cellulose fibres, which form a physical backbone for the flocs, was an essential constituent, since it ensured the formation of stable flocs, resulting in improved settling and dewaterability,

Dursun *et al.* (2004) formulated a synthetic surrogate for activated sludge using alginate, microcrystalline cellulose, yeast, Calcium, and Potassium ions. A cationic organic polymer was used as a conditioner. The synthetic sludge was considered to be an "adequate" surrogate for waste activated sludge. The rheological properties of both sludges were similar, with and without the addition of the conditioner. The filterability, as indicated by CST tests, the same type of response to polymer addition was seen. For research purposes, the synthetic sludge was considered to be acceptable for understanding trends in sludge behaviour; however, it did not exactly "duplicate" the properties of natural sludge. Nevertheless, since the relative trends in all of the measured properties were similar, the use of this type of synthetic sludge for experimental and modelling studies was validated.

Nguyen *et al.* (2006) compared the flocculation behaviour and dewatering properties of synthetic and activated sludge using a formulation similar to that described by Sanin and Vesilind (1996). The two types of sludge had similar settling and dewatering characteristics, and it was concluded that the synthetic sludge was a useful surrogate for activated sludge. Calcium ions were found to be important for the formation of flocs in both types of sludge. When no Calcium ions were added to the synthetic sludge, no flocs formed, and suspended particles were dispersed in the liquid solution; in contrast, in the activated sludge, some flocculation occurred in the absence of Calcium. Flocculation increased in both synthetic and activated sludge as the Calcium concentration was increased. Consequently the results of CST tests decreased with respect to an increase in the Calcium ion concentration. There were differences in the concentrations of Calcium required to promote flocculation in the two types of sludge. This difference was believed to be due to the absence of filamentous material in the synthetic sludge. It was therefore suggested that the addition of cellulose fibbers would make synthetic sludge a closer analogue to activated sludge. A similar conclusion was made by Sanin and Vesilind (1999) who considered that the main deficiency of synthetic sludge was the lack of filamentous microorganisms that form the backbone of the flocs in natural sludge. Subsequently, Nguyen *et al.* (2007) formulated a synthetic sludge containing Calcium, Alginate, and fibrous cellulose. The results of experiments indicated that the Calcium, Alginate, and fibrous cellulose had a significant effect on flocculation, and improved the properties of sludge as a surrogate for waste activated sludge.

The research discussed above considered only the use of synthetic sludge as a surrogate for waste activated sludge. In a review of methods for preparing synthetic sludge for laboratory testing, the European Committee for Standardization asked for the development of a standard synthetic sludge to be used in waste water management and academic research (Baudez *et al.*, 2007). Baudez *et al.* (2007) considered the formulation of both inorganic and organic sludges. Mixtures of Kaolin and calcite/quartz sand in water, at relative ratios ranging 90/10% to 75/25%, were proposed to simulate the behaviour of natural inorganic sludge.

1.3.3 The effects of filter papers on CST tests

The rate of flow of water through a filter paper which is permeable to water but not to solids varies with respect to numerous factors. Theoretically, according to Darcy's law, the main factors include the hydraulic permeability of the medium, the pore size of the filter medium, the area exposed to the flow, the dynamic viscosity of the fluid, the pressure difference between different parts of the filter, and the distance over which the pressure difference takes place (Dougherty and Franzini, 2007).

Several researchers (Hall and Hoff, 2002; Meeten and Smeulders, 1995; Lee and Hsu, 1994b) have described the processes involved in the dewatering of sludge on a filter paper. The force that drives the water to flow is the pressure difference across the sludge, generated by suction, either from external pressure or a compressed gas (e.g., in the SRF test) or by capillary action (e.g., in the CST test). During dewatering, the suspended particles move towards the filter, whilst the water also flows, relative to the particles, as described by Darcy's law (Smiles and Kirby, 1994; Hall and Hoff, 2002). A gradient develops in the particle concentration from the top to the bottom of the sludge, so that the hydraulic permeability of the medium, assumed to be constant in Darcy's law, varies with respect to the particle concentration gradient, and depends on the material balance within the particle distribution (Hall and Hoff, 2002). A combination of the concentration dependent assumption of Darcy's law with the material balance within the particle distribution controls the dewatering process, leading ultimately to the formation of a cake of sludge on the filter surface. When all the sludge solids are incorporated into the cake, there is an abrupt change in the flow of water. If no more water is available, the flow ceases (Hall and Hoff, 2002).

In a simple one-dimensional constant pressure system, the cumulative volume of water released from the sludge increases as the square root of the time (Hall and Hoff, 2002; Smiles and Kirby, 1994). This provides the underlying model for the CST test. As the water is expressed from the sludge, and moves outwards from the centre of the funnel to the edge of the filter, the CST changes as a function of the size of the wetted area on the paper. The flow of water is slowed down by both filter resistance and cake resistance, but in practice, the cake resistance is assumed to be much greater than the filter resistance (Lin and Lee, 2001). A controversy has arisen as to whether the filter paper is saturated, or unsaturated, and whether or not the flow of water is piston-like or diffusion-like. The piston-like model assumes that the paper is saturated, the invasion of water into the filter is a displacement process, and that capillary suction and permeability are constants. In contrast, the diffusion-like model assumes that the paper is unsaturated, whilst the capillary suction and permeability are not constants, but vary with respect to the water saturation profile of the paper (Lin and Lee, 2001). Nevertheless, both the diffusion-like and piston-like models yield the same outcome, that the CST is a geometric function of the size of the wetted area of the paper (Lin and Lee, 2001).

The factors that control the water flow in the filter during the CST test are not easy to measure or define, particularly if the filter papers are anisotropic, so the movement of fluid is not even across the whole diameter of the filter. Nguyen (1980) developed a theoretical model for the CST test assuming a perfect circle was formed by the filtrate front, so that the resistance to filtration was a function of the radius of the filtrate front as a function of time; this model, however, was too simplistic. Firstly, the model did not include sedimentation. Secondly, in practice, if the filter paper is anisotropic, the permeability and the capillary suction pressure along the grain of the filter paper are larger than those across the grain. Since the flow across the grain encounters a greater resistance than the flow along the grain, the filtrate moves faster along the grain. This means that at any given time, the radius of the front along the grain is greater than across the grain, and so the shape of the wet area is elliptical rather than circular (Lee and Hsu, 1994b; Lin and Lee, 2001; Tiller and Li, 2001).

Another factor of importance to CST tests is the effects of the interactions between particle-size and filter pore-size. If the sludge particles are similar in size to the pore sizes, then the CST will increase, due to the higher resistance to filtration associated with the blockage of the pores and cake formation. If the sludge particles are relatively larger than the filter pore size, a decrease in the CST will occur, due to the lower resistance to filtration associated with less blockage of the pores (Yukseler, *et al.,* 2007). Consequently estimates of CST will vary depending on the particle size composition of the sludge.

The paper originally used in the CST test devised by Gale and Baskerville (1967) was Whatman 17 chr. This is a smooth cellulose filter with a high flow rate, designed originally for paper chromatography. This type of anisotropic filter, although apparently selected at random by Gale and Baskerville (1967) has been applied universally ever since. Consequently, virtually all previous records of CST tests are based on the use of Whatman 17 chr papers. The use of the same types of filter papers, all of which are assumed to have the same, or at least similar, properties of permeability and pore size, has the advantage that the results of CST tests carried out by different workers are directly comparable. There are however, some concerns about the use of Whatman 17 chr since it is relatively more expensive than other papers, and due to its relatively large pore size, colloids may be lost from sludge suspensions through depth filtration (Scholz, 2005). In a preliminary study of CST tests using cheaper filter papers, with smaller pore sizes, Scholz (2005) suggested that if the sludge was stirred, to avoid sedimentation, then Fisher 200 chr, and possibly others papers, could be used to replace Whatman 17 chr. Scholz (2005) also recommended that further research should be undertaken to determine if other types of filter paper and porous filter media (e.g., cheaper, more isotropic, and with a different pore diameters) might be appropriate for use in CST tests, particularly for specific applications (e.g. different types of sludge). This suggestion provided a positive direction for the current study.

1.3.4 The effects of funnel geometry, stirring, and a sealant on CST tests

The standard funnel used in CST tests is circular. A 10 mm diameter funnel is generally recommended for light digested sludges but a larger 18 mm diameter funnel is advisable for heavy sludges, because it significantly reduces the time taken to conduct the test (Scholz, 2005).

The CST test times using circular funnels are generally higher than when using rectangular funnels (Lee, 1994a; 1994b). The reason for this difference is that a rectangular apparatus should theoretically overcome the problem of anisotropic filter paper by making use of the unidrectional flow in only one direction. Leu (1981) was the first to devise a CST apparatus with a rectangular funnel, in which the filtrate was restricted to flow only along the grain of the filter paper. The theory and application of using a rectangular funnel in CST apparatus was further developed by Unno *et al.,* (1983), Tiller *et al.* (1990), Tiller and Li (2001), Yukseler *et al.,* (1990) Lee (1994a, 1994b) and Lee and Hsu (1994b).

Tiller *et al.* (1990) and Tiller and Li (2001) modified the previous capillary suction model for use with rectangular funnels, taking sedimentation into account. A procedure was proposed to calculate the specific cake resistance from experimental data based on the new model. The analytical solution of the differential equation to describe the filtrate front travelling unidirectionally along the paper against time was presented, and this model was applied to estimate the accuracy of the specific cake resistance calculated experimentally. The specific cake resistance using the rectangular apparatus, taking sedimentation into account, was found to be about 40 times lower than when using the previous model.

Lee (1994a; 1994b) also formulated a model for CST tests using rectangular apparatus, assuming liquid flow in the paper was a diffusion process. When time was large, the relationship between the logarithm of the distance moved by the wet front and the logarithm of time was assumed to be linear, with a slope of $t^{1/2}$. The liquid saturation of the paper was modelled as a function of the sludge concentration, the specific cake resistance, and the paper thickness.

Lee and Hsu (1994b) considered that a rectangular funnel is superior to a cylindrical funnel for estimating the CST of heavy sludge with a high solid concentration, and a high specific resistance to filtration; however they considered the cylindrical funnel was a better choice when the solid concentration, sedimentation, and cake resistance was low. The theoretical reasons for this were explained by Chen *et al.* (1995) who expanded the dynamic model of Lee (1994b) to incorporate particle sedimentation. The conclusion was that sedimentation was the most influential factor determining the dynamics of capillary suction apparatus when the wet front was large. Particle sedimentation could, however, be neglected when the wet front was small. Consequently, a small circular funnel is adequate for CST tests using light sludges with a low cake resistance and low sedimentation, whereas a larger circular or rectangular funnels is better for CST tests using heavy sludges with a high cake resistance due to the influence of sedimentation.

It is clear from both theoretical and practical viewpoints, that one of the major problems of the standard CST test, particularly when testing heavy sludges, is that suspended particles accumulate on top of the paper by sedimentation. This may lead to an overestimation of the cake resistance, since the primary theory of the CST test does not take the effects of sedimentation into account (Bockstal *et al.,* 1985; Christiansen and Dick, 1985). Leu (1981) suggested that a flow of bubbles could be introduced into the CSA apparatus to reduce sedimentation. A constant current, induced by a stirrer within the sludge chamber, may also reduce or prevent sedimentation, thereby improving the results of CST tests (Scholz, 2005). Preliminary studies have indicated that there may be an interaction between the type of filter paper and stirring activity. Stirring was found to have a greater influence on the results of the CST test when using Whatman 17 chr than when using other papers such as Fisher 200 chr. It was proposed that the results of CST tests using Fisher 200 chr papers were not so negatively influenced by sedimentation as the results using Whatman 17 chr chr papers; however further research was considered necessary to expand on these preliminary findings (Scholz, 2005; 2006). This provided a direction and rationale for the current study.

Another issue associated with the use of CST tests, which has not been the subject of previous research, is the use of a sealant to close the gap between the metal funnel edge and the filter paper. The addition of a sealant should avoid leakage, which might be a source of variation in the results of replicated tests. It is hypothesized here that the addition of a sealant should improve the precision of CST tests, and research is required to test this hypothesis. Such a sealant should have specific properties. It should have high elasticity to close the gap between the metallic funnel edge and the filter paper without having to apply extra pressure to avoid blocking the pores of the filter paper at the point of impact between the funnel and the filter paper. The sealant should have insignificant overall thickness to avoid interference with standard testing conditions by not significantly altering the funnel volume and geometry. The sealant should be easy to fit to, and remove from the funnel and filter paper to avoid changing the funnel properties. Finally the sealant should have a low price and easy availability to avoid increasing the consumable costs of the CST test.

1.3.5 The rheology of sludges and the effects of temperature on CST tests

Rheology is the study of viscosity. Viscosity describes the resistance to flow of a fluid or suspension, and is a measure of its resistance to being deformed by stress. In Newtonian fluids, the shear stress is linearly related to the shear rate, so that viscosity should theoretically be constant for a particular fluid or suspension at a given particulate concentration and temperature (Dougherty and Franzini, 2007). The rheological properties of waste water sludges, however, cannot be easily described by simple deterministic models because they are non-Newtonian fluids. The viscosity of a given sludge sample is typically a non-linear function of its shear rate, and the effect of temperature on the viscosity of the sample varies with respect to the flocculation properties of the sludge. It is therefore virtually impossible to predict the viscosity of a sludge sample, given only its suspended solids content and the temperature (Dentel, 1997).

When the temperature is constant, the relationships between dewatering indices and the viscosity of sludges are complex and unpredictable. Dentel and Abu-Orf (1995) found that the CST increased non-linearly with respect to the suspended solids content and the viscosity of anaerobically digested waste water sludge. It was, discovered that the rate of mixing significantly influenced the dewaterability of the conditioned sludge by altering its rheological characteristics. Increased mixing led to a disruption of the flocs, causing an increase in the viscosity (i.e. a reduction in the fluidity) of the sludge with an associated increase in the CST (Abu-Orf and Dentel, 1999). When the influence of temperature is considered, the relationship between sludge viscosity and the results of CST tests become even more unpredictable.

Baskerville and Gale (1968) mentioned that the results of CST test were sensitive to variation in temperature. CST results tend to become lower with higher temperature, which is expected to be due to the decrease in filtrate viscosity (i.e. increased fluidity) with higher temperature. They suggested that to correct the CST values for testing temperature by using a correction factor of the ratio of the water at the testing temperature to a standard temperature. Despite this warning, there is no published research that described or predicted the effects of the variation in temperature on the results of CST tests.

It is apparent that the influence of temperature on sludge viscosity, and its assumed impact on the results of CST tests, requires more detailed consideration. The temperature dependence of viscosity is the phenomenon by which viscosity decreases as temperature increases. The relationship between viscosity and temperature can theoretically be modelled using the Arrhenius model (equation 1.3.1):

$$\eta(T) = \mu_0 \exp^{\frac{E}{RT}} \qquad (1.3.1)$$

where η = viscosity, T = temperature, μ_0 = a coefficient, E = the activation energy and R = the universal gas constant. This model predicts an exponential decrease in the viscosity with respect to the reciprocal of the temperature (Dougherty and Franzini, 2007). It has been found empirically that the relationship between the temperature and the apparent viscosity of a given type sludge, with known flocculation properties, at a given shear rate, can indeed be described by fitting an Arrhenius type equation to experimental data. For example, El Shafei *et al.* (2005) calibrated the Arrhenius equation with respect to the viscosity and the solid content of a sample of digested waste water sludge using the empirical model that was described in equation 1.3.2 below.

$$\eta = Ke^{1286/T} \qquad (1.3.2)$$

where η = viscosity; K = an empirical function of the solid volume fraction and T = the absolute temperature (ºC+273). The parameter *K* was defined empirically by equation 1.3.3.

$$K = e^{107C-9.1} \qquad (1.3.3)$$

where C = the volumetric fraction of solids.

These equations describe a non-linear decrease in viscosity with respect to a linear increase in temperature for digested sludge sample having a solid volume fraction of 0.01524 and a shear rate of 3.5 s^{-1}

The Arrhenius equation, however, does not completely explain the impact of temperature on the flocculation properties of sludge. Extracellular polymers, whose formation and structure is significantly affected by temperature, play an important role in determining the suspended solids content and associated dewatering properties of sludge. A high concentration of organic polymers in sludge significantly increases sludge viscosity due to an increase in the volume and strength of the flocs. Sludges with well flocculated particles have a higher viscosity than sludges with a low density of flocs, or sludges with disrupted flocs (Dick and Buck, 1985).

At higher temperatures, the rate of formation and the viscosity of polymers is decreased, whilst the hydrolysis of the polymers into soluble compounds increases (Jonsson and Jansen, 2006; Feng *et al.,* 2009). Thus higher temperatures may result in reduced flocculation, reduced settleability, and reduced viscosity, but an increase in the density of small suspended particles (Dignac *et al.,* 1988; Chundakkadu and Loosdrecht, 1999). For example, in a study of the effects of temperature and pH on the efficiency of flocculation and settlement of activated sludge, Ghanizadeh, and Sarrafpour (2001) found that when the temperature increased linearly from 15^{o}C to 35^{o}C (in 5^{o}C increments), there was a non-linear increase in the average suspended solids concentration from 43.3 to 67 mg/l, and a corresponding increase in the sludge volume index (SVI) from 40 ml/g to 130 ml/g. In contrast, the effects of increasing the pH of the sludge were the opposite of increasing the temperature. An increase in pH from 5.7 to 9.0 promoted flocculation, resulting in a decrease in the suspended solids content and SVI of the sludge. The implications are that sludge dewaterability, and hence the results of CST and SRF tests, may be significantly influenced by both pH and temperature, particularly when the pH is low, and the temperature is high, because the concentrations of suspended particles increases as a result of the reduced efficiency of flocculation.

It is evident, therefore, that this study, which focuses on the factors influencing the variability in the results of CST tests, must take the complex non-linear relationships between the temperature, the viscosity, and the flocculation properties of sludge into account as important controlling factors.

1.3.6 Modelling the results of CST and SRF tests

Three types of models can be constructed to describe systems of interest to engineers, which are classified as conceptual, mechanistic, or empirical (Svobodny, 1997; Ryan, 2007). A conceptual model is based on a hypothesis concerning the important factors that govern the behaviour of the physico-chemical processes and materials that comprise the system of interest. The disadvantage of conceptual models is that, although they provide a qualitative description of the characteristics and dynamics of a system, usually by means of diagrams, they do not mathematically define the materials and processes involved. A mechanistic model explicitly provides a deeper understanding of the physical and/or chemical properties of a system by describing, with mathematical symbols and equations, the theoretical or factual inter-dependencies between the materials and the processes. The disadvantage of mechanistic models is that they are usually deterministic, and describe only the ideal theoretical behaviour of a system, excluding random variation. Empirical models, on the other hand, do not necessarily describe a system qualitatively, theoretically, or deterministically. They are based on statistical relationships extracted from the variability that exists in real data e.g., by use of regression analysis. The advantage of empirical models is that they may reveal underlying links between apparently disparate variables, and they take random variation into account. The disadvantage of empirical models to engineers is that, although they may be useful for summarizing, describing, and predicting trends in the behaviour of physico-chemical systems in the real world, they are not necessarily mechanistically or theoretically relevant (Svobodny, 1997; Ryan, 2007).

Conceptual and mechanistic models of the materials (i.e., water and suspended solids) and the processes (i.e., filtration) involved in SRF and CST tests have previously been described (e.g., Ives, 1978; Meeten and Lebreton, 1992, Lee and Hsu, 1992; 1993; Meeten and Smeulders, 1995; Teoh *et al*., 2006; Yukseler *et al.,* 2007). When a suspension of particles in water (e.g., waste water sludge) is in contact with a filter, the filter retains the solids but allows the water (filtrate) to move. As more particles are retained, a cake builds up on the filter, and assists the filtration process. The rate at which the filtrate moves through the filter is assumed, according to Ruth's classical filtration theory to depend on the viscosity of the liquid, the thickness and resistance of the cake, and the pressure drop across the filter. (Dougherty and Franzini, 2007) It is based on the assumption that the average cake resistance is constant over time, and that the average values of porosity and pressure differential are also constant over time. Accordingly, the flow rate of the filtrate can be approximated by the differential equation 1.3.4 below.

$$\frac{\partial t}{\partial V} = \frac{\mu}{A(\Delta P)}\left(\frac{\alpha C V}{A} + R_m\right) \tag{1.3.4}$$

where t = time; V = volume filtered, μ = viscosity of filtrate, A = total area of filter, ΔP = pressure differential across the filter, C = concentration of solids per unit volume, α = specific resistance to filtration due to the cake (specific cake resistance), R_m = resistance to filtration due to the filter (filter medium resistance).

This equation can be re-written with symbols representing the larger terms as equation 1.3.5:

$$\frac{\partial t}{\partial V} = K_1 V + K_2 \qquad (1.3.5)$$

Where

$$K_1 = \frac{\mu \alpha C}{A^2 (\Delta P)} \qquad (1.3.6)$$

$$K_2 = \frac{\mu R_m}{A(\Delta P)} \qquad (1.3.7)$$

Theoretically, therefore, the plot of t/V versus V, where t = a given time and V = cumulative volume filtered, should be a straight line. The line has a slope K_1, equivalent to the average specific cake resistance (which is a measure of filterability or dewaterability) and an intercept K_2, equivalent to the filter resistance. This model is the basis for the extraction of a single value of the average specific resistance to filtration from the constant pressure filtration data obtained using the Buchner funnel test.

Classical filtration theory, however, has its shortcomings to describe the processes involved in sludge dewatering tests (Yukseler, *et al.,* 2007). For example, it assumes constant average filter porosity, constant average cake size, and constant pore-water pressure. In reality, the porosity and pressure differential across the cake, and the average cake size is not constant during the SRF test, particularly during the initial and final stages of dewatering, when the suspended solid concentrations are changing rapidly. A superior approach to modelling the filterability of sludge filtration systems was proposed by Yukseler *et al.* (2007). This approach was based on adapting models originally defined to describe the fouling of membranes, and corrected for the rapid increase in the slope of t/V versus V when the filter becomes blocked with particles and the flow rate of water slows down. It was suggested that the results of SRF test obtained during the blocking phase should be excluded, and that filterability is more accurately evaluated using a new parameter, called the cake filtration constant. The term t/V was substituted into the classical filtration model as follows (equation 1.3.8):

$$\frac{t}{V} = \frac{K_{CF}}{4J_o^2}V + \frac{1}{J_o} \qquad (1.3.8)$$

where K_{CF} = cake filtration constant, J = flow of filtrate, J_0 = initial flow of filtrate. Another important outcome of this research was the cake filtration constant was found to be sludge specific, i.e. it varied with respect to the quantity and quality of the suspended solids.

The model suggested by Yukseler *et al.* (2007) is not the only proposal for improving the analysis of the results of SRF tests. For example, Teoh *et al.* (2006) developed an alternative model based on the relationship between the specific cake resistance and the cake compressive stress.

In practice, the filterability of sludge varies with respect to many other factors including the particle size distribution of the sludge, the type of filter medium in use, the filtration area, and the mode of operation of the filter (whether down-flow or up-flow filtration). The pore size of the filter medium relative to the size of the particle size of the sludge solids is another major factor that controls filterability. Other factors which may influence sludge filterability include the concentration, particle charge, pH, organic content, floc density, mechanical strength, and cellulose content of the particles (Yukseler *et al.,* 2007). The assumptions of SRF models are also violated by the variable interacting effects of gravity, sedimentation, filtration, surface tension, and shrinkage (Christiansen and Dick, 1985; Bierck *et al.,* 1988).

Mechanistic models to describe the physical processes involved in the CST test have also been defined (Meeten and Lebreton, 1992; Lee and Hsu, 1992; 1993, Teoh *et al.*, 2006). These models also suffer from an inability to predict all the physical properties and processes that occur in reality, e.g., the sedimentation of the sludge, the flow of water through the filter, and the variable amount of water bound by the filter.

Several mechanistic models have been formulated to describe the transport of water in the filter paper during CST tests (Lee and Hsu 1992, Meeten and Lebreton 1992, Meeten and Smeulders 1995, Smiles, 1998, Lin and Lee, 2001). However, since there are variable forces associated with the movement of water through the paper, and the amount of water retained by the paper, definitive mathematical description of the process has not been formulated. The controversy as to whether the flow of water is diffusion like or piston like has not been entirely solved (Lin and Lee, 2001).

Few mechanistic models have been constructed to relate the SRF to the CST. Based on theoretical considerations and experimental data, Lee and Hsu (1993) proposed the model in equation 1.3.9:

$$\alpha_{av} = \left(P_{cd}(1 - s_{0,\infty})^{1.64}\right)\frac{2A^2}{C_0\mu}\left(\frac{dt}{dV^2}\right)_\infty \qquad (1.3.9)$$

where α_{av} = average specific resistance to filtration (m/kg); P_{cd} = capillary suction pressure, assuming a diffusion-like process, in which the paper is unsaturated; S_o = liquid saturation under the inner cylinder; A = cross section area (m^2); C_o = solid concentration (kg/m^3); t = time (s); μ= liquid viscosity (Pa.s); V = liquid invasion volume. When the wet front was large, and S_o was constant, the fluid flow across the cake in the CST test was assumed to be similar to a constant pressure filtration process.

The main practical disadvantage of the mechanistic models considered above is that they do not accurately predict the results of SRF and CST tests in practice, or the relationships between them. They do not take into account all the processes that cause variance in the test results. Variability, generally expressed in terms of the coefficient of variation (the ratio between the standard deviation of the CST and the mean CST) is a major issue encountered when performing multiple SRF and CST tests in practice (Scholz, 2005). Mechanistic models based mainly on theoretical considerations, cannot generally be applied to predict the variability in the results of CST and SRF tests experienced in reality. The rationale for the current modelling study, therefore, was not based on the need to develop mechanistic models, but on the need to construct empirical models to describe the variability in the results of CST and SRF tests performed experimentally. Such models have to be calibrated using real experimental data, to take into account the residual variance around the mean estimates of CST and SRF, which conceptual and mechanistic models are unable to define. To date, few such models have been formulated, and research on this topic is limited, providing a positive direction for the current study.

Chapter Two

General Methodology

2.1 Choice of sludge

The common application of the CST apparatus is to evaluate the dewaterability of sludge from wastewater treatment plants. Sludge samples were therefore chosen for the experiments undertaken in this study. Different types of natural sludge, and a synthetic sludge were used for testing purposes.

2.1.1 Natural sludge

Samples of primary sludge (called Primary 1 and Primary 2) were obtained from a Scottish Water wastewater treatment plant in North Queensferry, near Edinburgh, UK. The samples were drawn from holding tanks, which collect sludge from primary settlement tanks and septic tanks. Typically, primary sludge contains sedimentable solids from wastewater, and has a high organic content originating mainly from fecal matter and food scraps. Primary sludge is a typical representative of heavy sludges (sludge with CST value higher than 50 seconds using Whatman filter paper and 18mm circular funnel was considered as heavy slow filtering sludge in this study). The samples were stored at $4^{o}C$ in a refrigerator, constantly aerated and used for testing within 4 days.

Samples of surplus activated sludge (called Surplus 1 and Surplus 2) were collected from the same waste water treatment plant as the primary sludge. The samples were taken from the final sedimentation tanks of the activated sludge process. Surplus activated sludge usually contains light flocculent biological solids, and is a typical representative of light sludge from secondary sedimentation processes (sludge with CST value lower than 50 seconds using Whatman and 18mm circular funnel was considered as light fast filtering sludge in this study). Activated sludge samples usually have higher moisture content in comparison with primary sludge types, and they are therefore associated with greater dewaterability problems. The CST tests with surplus activated sludge were performed within two days of sampling. Meanwhile, the samples were stored at 4^{o}C and constantly aerated in a refrigerator.

It was a novel application in this study to test the dewaterability of sludge taken from wet gully pots (called Gully Pot1 and Gully Pot2). This type of heavy sludge is increasingly becoming a costly problem for many local authorities, and treatment costs can be reduced by optimizing sludge dewatering (Scholz, 2004). The gully pot sludge contains both organic and inorganic matter, and its composition varies greatly in space and time. The samples were strained with a sieve of 250 µm diameter pore size to simulate preliminary treated wastewater. They were subsequently stored at 4^{o}C, continuously aerated and used for testing within 5 days.

The physical properties of the different types of natural sludge used for testing are presented in Table 2.1.1

Table 2.1.1 Typical properties of natural sludges

Sludge	Total suspended solids (g/l)	pH
Gully pot sludge	412-424	6.0-7.5
Primary sludge	28-34	6.4-6.5
Surplus activated sludge	2-8	7.0-7.5

2.1.2 Formulation of synthetic sludges

The natural sludges routinely used for CST tests in practice are unstable because their physical and chemical properties are influenced by the metabolic activities of living microorganisms. Inconsistencies in the results of CST tests may therefore depend on the ages and biological activities of the sludge samples (Örmeci and Vesilind, 2000). The development of synthetic sludges with similar dewatering properties to those of natural sludges, but with more consistent CST test results, was crucial to enable efficient testing of modifications to the CST devise and methodologies. Moreover, the use of synthetic sludges permitted experiments to be performed over a long period of time using samples with similar properties.

Different old and novel recipes for synthetic sludge that were potentially useable as a surrogate for natural sludge were considered, and tested to overcome the problem of the time-varying characteristics and properties of natural sludges. Synthetic sludges with different dewatering properties to simulate different sludge types were formulated by changing their ingredients (Dursun *et al.,* 2004). The purpose was to formulate synthetic sludges with CST values of similar magnitude in comparison to natural sludges. The synthetic sludge was designed to resemble the physical properties (i.e. rheology and dewaterability) of typical sludge flocs and the behaviour of activated sludge, by conditioning with a cationic synthetic polymer (Örmeci and Vesilind, 2000). The dispersion of flocs determines the density, particle size and distribution, and porosity of activated sludge flocs. These properties determine the distribution of water in sludge and, in turn, properties such as filterability, settleability and rheology (Sanin and Vesilind, 1999). However, it was unjustifiable to reproduce the recipes of some synthetic sludges described in the literature due to the unavailability and/or high costs of the ingredients.

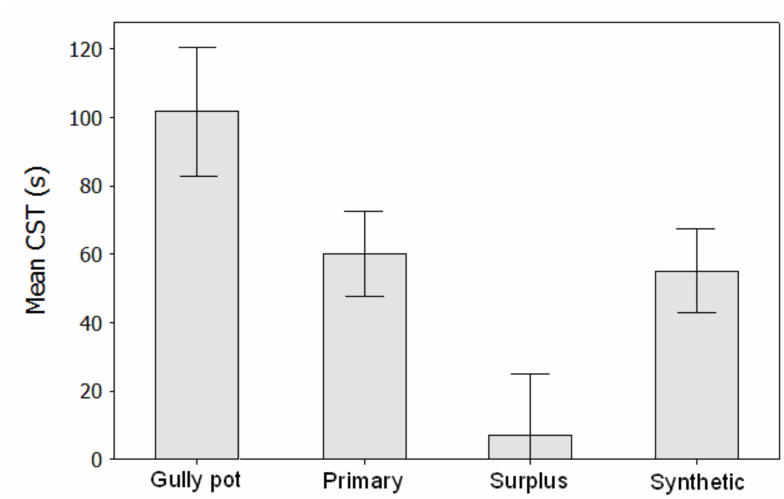
The selected synthetic sludges were based on mixtures of clay slurries, which are the most common constituents for the production of stable and reproducible synthetic sludge. Different concentrations of two types of clay (i.e. Kaolin and Bentonite) were tested for their CST values. The cohesive property of Kaolin clay particles is primarily due to their edge-to-face electrostatic alignments. Bentonite clay undergoes osmotic swelling when exposed to moisture, and its shear strength is mainly due to van der Waals attraction forces and partly due to inter-particle friction. Kaolin was found to be adequate to simulate individual bacteria (Chu and Lee, 2005). In addition to Kaolin and Bentonite clay, which emulate bacteria, cellulose fibres can be used as substitutes for filamentous micro-organisms, Sodium alginate to simulate microbial extra cellular polymers and Calcium ions as surrogates for bridging cations. Calcium ions have a high affinity for an extra-cellular polymer structure, and Calcium is involved in bioflocculation (Sanin and Vesilind, 1999).

The first mixture contained 100 ml distilled water and 10% w/w Kaolin. Mixture 2 contained 100 ml distilled water and 2% w/w Kaolin. The third mixture was created by mixing 100 ml distilled water with 1% w/w Bentonite. Mixture 4 contained 100 ml distilled water, 7% w/w Kaolin and 3% w/w Bentonite. Mixture 5 was made up of 100 ml 85 mM Sodium chloride solution, 0.1% w/w Kaolin clay, 10 mg/100 ml Sodium alginate, 60 mg/100ml cellulose fibres and 548 mg/100ml $CaCl_2$ $6H_2O$. Mixture 6 contained 100 ml 85 mM Sodium chloride solution, 5% w/w Kaolin clay, 10 mg/100 ml Sodium alginate, 60 mg/100ml cellulose fibres, 548 mg/100ml $CaCl_2$ $6H_2O$. Finally, Mixture 7 was made up of 100 ml 85 mM Sodium chloride solution, 3.33% w/w Kaolin clay, 1.67% w/w Bentonite clay, 10 mg/100 ml Sodium alginate, 60 mg/100ml cellulose fibres and 548 mg/100ml $CaCl_2$ $6H_2O$. Using combinations of the above ingredients, the seven different synthetic sludge mixtures were designed and tested. Primary results showed inconsistency of CST values for most of the tested mixtures. Synthetic sludges, which were not homogenous, were discarded. The most suitable synthetic sludge used was Mixture 7 as it was homogeneous and produced more consistent results.

The first five mixtures were prepared by suspending Kaolin and/or Bentonite in distilled water and stirring the ingredient(s) until they became homogeneously mixed. Mixtures 6 and 7 were prepared as described elsewhere (Örmeci and Vesilind, 2000). Kaolin and Bentonite were mixed and then suspended in 85 mM Sodium chloride solution. Cellulose fibbers and Sodium alginate were added and the suspension was stirred for 3 hours. During this time, the Alginate was adsorbed. Afterwards, Calcium was added to the mixture, and the suspension was considered fit for purpose when the flocculation process was finished. The sludge was thoroughly mixed before each use to make sure the suspension could be regarded as homogenous to all tense and purposes.

Comparing the CST test values of different types of natural sludge with synthetic sludge (mixture 7) using the standard CST apparatus indicated that this mixture was most effective in simulating the dewatering properties of primary sludge, because the mean CST values were similar (Figure 2.1.1).

Figure 2.1.1 Comparison of the mean CST estimates ± 95% confidence intervals of Gully pot, Primary, Surplus activated, and Synthetic (mixture 7) sludges



Further investigations were carried out to develop synthetic sludge with higher CST values than mixture 7, to simulate the CST test results for waste activated and digested sludges. Physical and chemical tests and rheological measurements were made on several mixtures of the synthetic sludges and also on natural sludges. The final recipe of the synthetic sludge used in this study was based on the method described by Dursun *et al*., (2004). The same procedure was followed, but Kaolin and Bentonite were used instead of fresh yeast. The synthetic sludge contained Sodium alginate to simulate microbial extracellular polymers. Calcium ions were used as bridging cations. The Calcium and Alginate concentrations were chosen to simulate common ranges of microorganisms that normally exist in the wastewater treatment sludge (Sanin and Vesilind, 1996). Bentonite and Kaolin were applied as surrogates to simulate individual bacteria (Chu and Lee, 2005). Cellulose fibres were used to simulate filamentous microorganisms, which are considered as the back-bone of the sludge flocs.

The physical and chemical properties of the developed synthetic sludges, specifically their viscosity, pH, suspended solids and supernatant turbidity, were measured and compared with the properties of the primary, activated, waste activated and digested sludge obtained from Seafield Waste water treatment plant, Edinburgh, UK. Viscosity is one of the most important characteristics of sludges, because it defines the deformation of sludge components under the influence of stress (Barnes *et al.,* 1993). An understanding of sludge viscosity is important in management operations, e.g., estimating the design parameters of transportation and pumping schemes, determining the sludge network strength to evaluate sludge conditioning and to improve dewatering properties (Abu-Orf *et al*., 2003; Abu-Orf and Örmeci, 2005) and estimating the quality of flocculated sludges (Tixier *et al*., 2003). Due to the non-Newtonian nature of sludge flow, the shear stress is not linearly related to the shear rate, so that the sludge viscosity depends on the shear rate gradient at specific conditions of pressure and temperature (Seyssiecq *et al*., 2003; Chen *et al*., 2005).

A synthetic sludge to simulate activated sludge was formulated using 5 g Sodium alginate dissolved in 1000 ml of distilled water. 6 g of cellulose fibres were added and subsequently mixed to create a suspension. 23.3 g of Kaolin and 11.7 g of Bentonite were mixed together and added slowly to the suspension by gentle stirring for 120 min at 10 rpm to allow for the incorporation of alginate into particle surfaces (Dursun *et al.,* 2004). Subsequently, 250 ml of 146 g/l potassium chloride solution was added and stirred to suspension at 10 rpm for 2 min. Furthermore, 250 ml of 5 g/l Calcium chloride was added and stirred at 10 rpm for 3 min. This synthetic sludge was considered to be too concentrated, and was therefore diluted with distilled water to several ratio or percentages. This dilution also reduces the salinity, which is known to affect the SRF and consequently the CST (Lai et al. 2004). Five different concentrations of synthetic sludge were formulated (Table 2.1.2) by mixing different parts of concentrated sludge with distilled water.

Table 2.1.2 Concentrations of synthetic sludges

Synthetic sludge concentration	Parts of concentrated sludge	Parts of distilled water
10%	1	9
20%	2	8
30%	3	7
40%	4	6
50%	5	5
100%	1	0

2.1.3 The physical and chemical properties of synthetic sludges

The physical and chemical properties of different concentrations of synthetic sludge were investigated. These properties included the TSS (total suspended solids), the pH measured by HANNA HI 991300 and the turbidity of the supernatant, measured using HACH 2100N after 30 min of settling. The tests were based on standard methods for examination of water and waste water (American Public Health Association, 1998). Viscosities were measured with an AR 2000 rheometer with concentric cylinder geometry. The concentric cylinders had a 500 μm double gap rotor with 20.00, 20.38 and 21.96 mm stator outer, rotor inner and rotor outer radii, respectively. The properties of different synthetic sludge concentrations were compared to those of the natural sludges to determine the most appropriate synthetic sludge that simulated natural sludge, particularly waste activated and digested sludge.

Desorptivity characterises the water retention properties of sludges. When the loss of water is controlled by capillary suction the amount of water initially increases proportionally to the square root of time, until a certain point, when it tends towards an asymptote. Desorptivity was estimated from the slope of the line between the cumulative desorbed volume of water per unit area versus the cumulative desorption time during the initial stage of water loss:

$$i = R\sqrt{t}$$

where i = cumulative desorbed volume of water per unit area (mm = mm^3/mm^2); t = cumulative desorption time (min); R = Desorptivity ($mm.min^{0.5}$) A low value of R indicated that the sludge exhibited high water retention ability, whereas a high value of R indicates the sludge exhibited low water retention ability. Consequently, the CST estimates were expected to decrease when the desorptivity of the sludge increased.

The desorptivity of synthetic sludge samples was estimated at sludge TSS concentrations of 2.3, 5.64, 8.8, 12.1, 15.3 and 31.6 g/l and temperatures of 10, 15, 20, 25 ^{o}C using standard CST test apparatus (Whatman 17 chr filter paper and circular funnel) Desorptivity was estimated as the reciprocal of the square root of the slope of the cumulative desorbed volume of water versus the desorption time.

It was important to measure the floc particle size distributions in the synthetic sludges formulated for the purposes of this study. Particle size distributions influence the physical and chemical properties of sludges, including viscosity, settleability, compressibility, aggregation behaviour and the transfer of minerals between flocs (Chaignon *et al.,* 2002; Jin *et al.,* 2003). The floc size distributions of sludges vary with respect to the presence of conditioners, and also to shear stress, which induce flocculation, depending on the conditions of agitation and dilution (Spicer *et al.,* 1998; Maggioris et al., 2000; Nguyen *et al*., 2007; Shatat *et al.*, 2008; Nguyen *et al.,* 2008). The different physical configurations and hydrodynamics of waste water treatment plants also influences the floc size distributions of sludges (Jin and Lant, 2004). The potential influence of variations in particle size distribution on the results of CST tests (Scholz, 2005; Yukseler, *et al.,* 2007) was the main reason for including a particle size distribution analysis of the synthetic sludges used in this study. A floc particle size distribution analysis was performed using different formulations of sludge. The relative volumes of floc particles of different sizes in synthetic sludge samples containing different ingredients were estimated using the image analyzing device Malvern Master Sizer Laser Radiation class 3B laser product (max. output 5 mWCW He-Ne 632.8 nM; particle size detection range: 0.05-880 μm).

2.2 The estimation of CST and SRF

2.2.1 The CST test

Two patented CST device (Model 304B CST and Model 319) provided by Triton Electronics Ltd. were used to conduct all the experiments described in this thesis (Figures 2.2.1 and 2.2.2). The single radius Model 304B device is small in size and battery-operated, which therefore makes it portable and handy for onsite testing. The device consists of a cylindrical steel funnel resting on a Whatman 17 chr filter paper fitted between two Perspex plates with electrode sensors across the top plate.

The electrodes are placed at standard interval distances apart from the centre of the funnel, to ensure a constant volume between each electrode, and are connected to a timer. The electrodes are used to measure the travel times of the water front. The recorded CST values (in seconds) are automatically displayed on a screen. They provide a measure of the time required for the water front to move through the areas of paper positioned between the electrodes.

The multi radii CST device Model 319 can produce five CST values (CST1, CST2, CST3, CST4 and CST5) instead of one CST value. The CST value from the standard device (single radius CST device Model 304B) is equivalent to the second CST value of the multi –radii CST device Model 319.

Each CST test was executed by pouring an adequate and representative amount of suspension of a homogenous sludge sample into a funnel until the liquid was level with the top rim of the funnel. The capillary suction generated by the pores of the filter paper forced the filtrate to be sucked from the suspension at the point of contact with the paper. A cake remained as a residue on top of the paper. The capillary suction pressure of the porous paper is about twice as large as the hydrostatic pressure head within the funnel. Therefore, it can be assumed that the CST test value is independent of the quantity of the liquid in the funnel, as long as there is sufficient liquid to generate the suction pressure (Meeten and Smeulders, 1995).

The filtrate moved across the filter paper and timing started when the wet front of the filtrate reached the starting electrode placed at 1.59 cm from the funnel centre. The time required for the wet front to reach each of the five electrodes was automatically recorded by the device producing five CST recordings in the multi radii CST device model 319.

Figure 2.2.1 Standard apparatus (Model 304B CST) to measure the CST (CST) at one fixed position

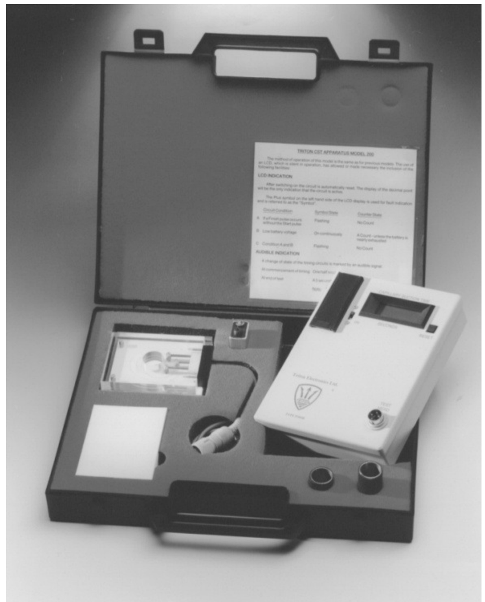


Figure 2.2.2 Multi Radii CST device (Model 319) to measure five CST values

The device model 319 used in this study (section 4.4, chapter five and six) produced five CST values (CST1, CST2, CST3, CST4 and CST5) instead of one CST value, as generated by a standard single radius CST device. The CST value from the standard device is equivalent to the second CST value of the multi-radii device. The second value was used throughout this study when a single estimate of CST was required. The five CST values were regressed on the distance between each electrode to the power of four to estimate filterability and SRF.

2.2.2 The estimation of SRF using the CST test

It is reported that, for a wide range of different types of sludge, there is a correlation between the SRF, estimated using the Buchner funnel test, and the rate at which the water travels between the electrodes of the CST device (Scholz, 2005). However, this relationship has not yet been fully described in statistical terms, providing a direction for the current study.

Filterability estimates were computed from the results of the CST tests. The slope of the five values of CST (CST1, CST2, CST3, CST4 and CST5) regressed on the distance between each electrodes to the power of four (where the distance was measured between the starting and stopping electrodes) was assumed to provide an estimate of filterability, following Meeten and Smeulders (1995). Four different linear regression equations were evaluated to discover the best line of fit, and thereby extract the most accurate and precise filterability estimate from the results of each CST test. The reason for using four different lines was that the slope of a regression line varies in magnitude, depending on whether or not intercepts are fitted, and whether or not the variables have been transformed. The presence or absence of intercepts and the use of transformations were therefore taken into account when evaluating the use of the slopes of the CST measurements versus the distance between the electrodes as estimates of filterability.

The CST test was performed at three temperatures 15^{o}C, 20^{o}C and 25^{o}C for four total suspended solid (TSS) concentrations (8.8, 12.1, 15.3 and 31.6 g/l). Consequently, there were twelve experimental treatments (three temperatures times four TSS concentrations). Five replicates for each treatment were used to take the variance of the estimates into account. Therefore, a total of 60 samples were analyzed. Simple linear regression models were constructed using data from each treatment to define the relationships between CST and the distances between the electrodes in the CST device (Section 6.1). Meeten and Lebreton (1992) defined this relationship in terms of a mechanistic model, which assumed linearity between CST and distance to the power of four (distance^4) between the electrodes. The slope of this line was taken as an estimate of filterability, which was assumed to be a function of SRF. Four different linear regression equations for each treatment, named A, B, C and D were evaluated to discover the best line of fit, and thereby extract the most accurate and precise filterability estimate from the results of each CST test. The reason for using four different models was that the slope of a regression line varies in magnitude depending on whether or not intercepts are fitted, and whether or not the X and/or Y variables have been transformed (Ryan, 2007). The presence or absence of intercepts and the use of data transformations had to be taken into account when evaluating the use of slopes to estimate the best possible filterability estimates using the results of the CST tests. The four models are defined in.

$$Y = \beta_0 + \beta_1 X \pm \varepsilon \qquad \text{(Model A)}$$

$$Y = \beta_1 X \pm \varepsilon \qquad \text{(Model B)}$$

$$\log_e Y = \beta_o + \beta_1 \sqrt{X} \pm \varepsilon \qquad \text{(Model C)}$$

$$\log_e Y = \beta_1 \sqrt{X} \pm \varepsilon \qquad \text{(Model D)}$$

The variables in models are Y=CST (measured in seconds); β_0 is the intercept (magnitude of CST when distance[4] between the electrodes of the CST device is zero), β is the slope (assumed to be a function of SRF, and termed filterability), X is the distance[4] between the electrodes of the CST device; ± ε is the residual error either side of the best line of fit (i.e. the differences between the predicted values of Y and the observed values of Y).

It is standard practice in regression analyses to estimate both the slope β and the intercept β_0 simultaneously (and also to test if the intercept and the slope are significantly different from zero at a prescribed significance level (Chatterjee *et al.,* 2000), conventionally $\alpha = 0.05$. However, a regression equation can also be constructed by not fitting an intercept, when β_0 is assumed, for analytical or technical reasons, to be zero. The magnitude of the intercept when estimating filterability as the slope of a regression line was an important issue, because it is debatable if the CST is zero in practice, if the distance[4] between the electrodes is zero. The mechanistic model infers that the intercept is theoretically zero, but it is possible that due to the high absorption rate at the beginning of the test (Hall and Hoff, 2002), leakage of filtrate between the filter paper and the funnel causing surface flow of the filtrate, or due to unknown experimental error and/or technical reasons that the intercept is not zero. For example, if there is a leakage and surface movement, then the straight line plot of CST on distance^4 could be displaced at the origin. Consequently, it is necessary to determine whether an intercept should be included when estimating filterability based on the slope of the regression line of CST on distance^4. Consequently, linear regression analysis was performed twice, assuming that β_0 is not zero (so that both the slope and intercept were estimated simultaneously) and assuming that β_0 is zero (so that only the slope was estimated, without fitting the intercept).

Two linear regression models (Model A with a fitted intercept and Model B without a fitted intercept) were constructed using untransformed data, and two models (Model C with a fitted intercept and Model D without fitting an intercept) were created using transformed data. The reason for transforming the data was to determine if the goodness of fit of the linear relationship between CST and distance^4 could be statistically improved e.g., by reducing or eliminating violations of the theoretical assumptions of regression (Chatterjee *et al.,* 2000). This is important analytically, because deviations from residual normality and non-homogeneity of variance could influence the accuracy and precision of estimates of filterability based on the slopes of CST on distance^4. Therefore, transformations were applied to both X and Y variables in an attempt to eliminate or reduce violations of the theoretical assumptions of regression. Such violations control the magnitudes of the regression coefficients and their standard errors (Ryan, 2007). The residuals (differences between the observed Y values and the Y values predicted by a regression equation) should ideally be normally distributed, dispersed randomly and evenly either side of the regression line, and have a mean of zero. Ideally there should be no outliers; i.e.

extreme residuals, which cause the relationship to deviate from the linear trend, and/or which indicate that the variance in the Y variable around the best line of fit is not equal (homogeneous) for all values of the X variables. Transformations (e.g. using logarithms, roots, or exponents) are used routinely in regression analysis to linearize the relationships between Y and X, to normalize the residuals and to homogenize the variances (Ryan, 2007). In this investigation, deviation from normality and homogeneity of variance could potentially influence the accuracy and precision of estimates of filterability using the slopes of CST on distance[4]. Some researchers simply choose the best transformation for regression analysis by trial and error (Tabachnick and Fidell, 2007). Logarithmic and square root transformations in Models C and D were, however, justified for the purposes of this investigation by the application of the Box-Cox tests. A Box-Cox test is an exploratory tool which uses a maximum likelihood method to determine the optimum transformation parameter. A control chart with an optimized estimate for λ (the transformation parameter) denoted as "Lamda". Although the optimum estimate of λ is a specific number, in a practical situation the selected value of λ should correspond to a standard transformation that is easy to interpret; e.g., square root (λ=0.5) or logarithm (λ=0). In practice, a logarithmic transformation is performed when the estimate of λ is close to 0, the lower and upper limits of the 95% confidence interval approach zero, and the rounded value of λ=0.00. A square root transformation is performed when the estimate of λ is close to 0.5, the lower and upper 95% confidence limits approach 0.50, and the rounded value of λ=0.50. The reason why logarithmic and square root transformations improve the fit of linear regression models is that they correct for the right-skewed distributions of residuals. A logarithmic transformation, however, may over-compensate a right-skewed distribution and create a left skew. In such a case, a square root transformation, which has less impact on right-skew, is the optimum transformation, in preference to logarithms (Tabachnick and Fidell, 2007). Consequently, both logarithmic and square root transformations were used to construct Models C and D. The transformation parameter for the X variable ($distance^4$) was kept unchanged at λ=0.50 for the 12 treatments, implying that a square root transformation was generally appropriate. The transformation parameter for the Y variable (CST), however, was predominantly λ= 0.00 implying that a logarithmic transformation was appropriate. Consequently, it was decided to perform a square root transformation of the X variable ($distance^4$) and logarithmic transformations of the Y variable (CST). Since four models were applied, linear regression analysis generated four slopes (filterability estimates) for each treatment, denoted A, B, C and D as follows: the filterability computed from Model A was the slope of CST on $distance^4$ assuming the intercept was not zero; the filterability computed from Model B was the slope of CST on $distance^4$ assuming the intercept was zero; the filterability computed from C was the slope of $\log_e$ CST on assuming the intercept was not zero; and the filterability computed from Model D was the slope of $\log_e$ CST on assuming the intercept was not zero.

2.2.3 The SRF test

To analyze the relationships between the results of CST and SRF tests, estimates of the specific resistance to filtration (SRF) were made at the same conditions used for the CST tests. Each SRF value was estimated using a Buchner funnel, with using synthetic sludge samples, applying 0.65 bar vacuum suction pressure, using Whatman No. 2 as a filter medium. After applying the suction pressure the volume of the filtrate was collected as a function of time. Four different solid contents of synthetic sludge were used (8.8, 12.1, 15.3 and 31.6 g/l) and the tests were run at three different temperatures using a controlled temperature room (15^oC, 20^oC, and 25^oC). Each treatment was replicated five to sixteen times. The SRF values were estimated using the classical filtration model. SRF was estimated as the slope of the line of t/V versus V where t = a given time and V = cumulative volume filtered. The mean SRF (±standard deviation) and the 95% confidence intervals were computed for each treatment. At the duration of each filtration experiment, the solid content of the filter cake was measured by drying the filter paper (Whatman No.2) in the oven at 105°C for 24 hours. The difference between the weight of the wet filter cake and the dry filter cake on the filter paper was calculated, and taken as the dry solid content of the filter cake. The viscosity of the filtrate was measured using AR 2000 rheometer with concentric cylinder geometry. The concentric cylinders had a 500 μm double gap rotor with 20.00, 20.38 and 21.96 mm stator outer, rotor inner and rotor outer radii, respectively. In this study, a model to predict the relationships between CST (filterability), TSS, Temperature, and SRF will be developed based on empirical data.

Various potential types of non-linear models were explored in this study to predict the results of SRF tests from the results of CST tests. Non-linear behaviour was described by empirical multiple regression models, which included reciprocal, logarithmic, squared or cubic transformations of the variables (Section 2.9).

2.3 The effects of the filter papers on the CST test

The standard paper commonly used with the CST apparatus is the Whatman 17 chr paper (Whatman Plc., Brentford, UK), which was considered as the benchmark paper for the purpose of this research paper. The Whatman 17 chr paper has been used since the invention of the CST apparatus. It is a chromatographic paper made of cellulose with a high flow rate of 190 mm per 30 min and with a mean pore diameter of 8 μm. However, the paper has some disadvantages in the context of the CST test including its anisotropic properties and relatively over-sized pores.

Therefore, alternative cheaper filter and chromatographic papers from different sources (Fisher 200 chr manufactured by Fisher Scientific Ltd., Loughborough, UK, and distributed by Camlab in the UK; HOVO TO w/s from Hollingsworth and Vose Ltd., Kentmere, UK; Carlson EE1.OH from Carlson Filtration Ltd., Barnoldswick, UK; SS 1107, SS 3205 and SS 3324 chr from Schleicher and Schüll Microscience GmbH (part of the Whatman Group since 2004), Dassel, Germany; MN 440 B and MN 280 from Macherey and Nagel GmbH, Düren, Germany; FN 30, BF 4, BF 3 and FN 8 from Munktell and Filtrak GmbH, Germany) were also tested, and subsequently compared with the Whatman 17 chr paper. Some of the papers used were isotropic, validated with ink drop tests (e.g., HOVO TO w/s, Carlson EE1.OH, SS 1107 and SS 3205). The paper properties and purchasing costs, volunteered by the manufacturer and/or supplier are summarized in Table 2.3.1.

Table 2.3.1 Summary of filter paper properties and costs

Paper	Type	Flow rate (mm /30 min)	Basis weight (g/m^2)	Thickness (μm)	Pore diam -eter (μm)	Isotropic	Cost per CST paper (£)
Whatman 17 chr[a]	chr[a]	190	413	920	8	No	0.14
Fisher 200 chr[a]	chr[a]	180-220	440	900		No	0.07
HOVO TO w/s			280		6.7	Yes	0.01
Carlson			425-475	800-1200	5-6	Yes	0.02
SS1107		121	140	280	4-7	Yes	0.02
SS 3205		136	95	200	6-12	Yes	0.01
SS 3324 chr[a]	chr[a]	180	280	730		No	
MN 440 B	chr[a]	130-145	400	1000		No	
FN 30	chr[a]	240	320	900		No	0.09
BF 4	BP[b]		550	1300		No	0.15
BF 3	BP[b]	170	280	500		No	0.09
FN 8	chr[a]		330	760		No	0.07

[a] Chromatographic paper; [b] Blotting paper.

2.4 The effects of the funnel geometry on the CST test

Two different sizes of cylindrical funnels were tested: a 10 mm diameter funnel with a volume of 5.62 cm^3 for light and fast filtering sludge, and an 18 mm diameter funnel with a volume of 6.36 cm^3 for heavy and slow filtering sludge. A rectangular funnel was used to overcome or at least reduce the negative effect (i.e., uneven water distribution) of the anisotropic property of many papers including the standard Whatman 17 chr paper. The rectangular funnel was designed to have a similar volume in comparison to the 18 mm cylindrical funnel. Dimensions of 18 mm width, 18 mm length and 20 mm height were therefore chosen. The rectangular funnel was tested with both light and heavy sludge, and corresponding results were compared with data obtained with both types of cylindrical funnels. The standard contact area between liquid and paper was enlarged to fit the rectangular funnel, but the probes were placed at the same positions as with the circular funnel. The tests with the different funnel geometries were carried out using all different types of test paper, if it was applicable.

Further experiments were carried out to investigate in more depth the effect of the funnel geometry on the water front movement through the filter paper and to understand the differences in the CST values when circular and the rectangular funnels were used. The experiments were done by recording the time versus the distances of the water front through Whatman 17 chr filter paper for both circular (with and without using the sealant) and the rectangular funnel (with and without using the sealant) using distilled water. For the circular funnel regular size of Whatman 17 chr filter paper which used in the standard CST test was used (70×90 mm). The circular funnel was placed at the centre of the filter paper. The filter paper was marked at five regular distances from the centre of the circular funnel. The time needed by the absorbed water front to move through the filter paper was recorded. The experiments were repeated five times for both the circular and the rectangular funnels with and without sealant (n = 20 runs in total).

Experimental data were fitted based on the general model as in equation (2.4.1) (Hall and Hoff, 2002). This model describes the movement of the water front through porous media taking into consideration the effect of the funnel geometry.

$$i = a + St^{1/2} + GS^2t \qquad (2.4.1)$$

where i = the cumulative absorbed water volume; a = intercept constant of the model; S = the sorptivity, t = the time; and G = the geometrical factor. The experimental data of circular funnel were fitted to the model (equation 2.4.2) which is a special case of the equation 2.4.1 when cylindrical source is used (Hall and Hoff, 2002).

$$i = St^{1/2} + \frac{1}{3}\frac{S^2}{f_e r_o}t + ... \qquad (2.4.2)$$

where f_e = the open porosity of the porous media; r_o = the radius of the cylindrical source; and i for the cylindrical source = $\frac{f_e(r^2 - r_o^2)}{2r_o}$. The experimental data of the rectangular funnel were fitted to equation 2.4.3 which is special case of equation 2.4.1 when a strip source is used (Hall and Hoff, 2002).

$$i = St^{1/2} \qquad (2.4.3)$$

This model describes the movement of the water front through the porous medium taking into consideration the effect of the funnel geometry. For the rectangular funnel, the same procedure was followed, but a strip of Whatman 17 chr filter paper was used instead of the regular size. The size of the strip was 24×90mm. The width of the strip of the filter paper is the width of the rectangular funnel + 2mm from each of the lateral sides of the rectangular funnel which was placed in the middle of the filter paper strip.

2.5 The effects of stirring on the CST test

The CST device was altered by the incorporation of a stirrer into the design. The purpose was to evaluate the possible influence of sedimentation on the CST values. The key objective was to quantify the corresponding effect on the mean and variability of CST results. The stirrer was adapted from a toy manufactured by K'nex. The toy motor is battery-operated, and drives an L-shaped stirring shaft. The horizontal part of the shaft is 6 mm long and 3 mm in diameter. The maximum number of rotations per minute is only 35. Experience indicated that reduced stirrer speed help to avoid the destruction of weak sludge flocs. The deviation of the shaft from the centre during the rotation was observed to be < 1 mm. The stirrer was only used with heavy and slow filtering sludge types. Therefore, the stirrer was only applied using the 18 mm diameter circular and the rectangular funnels. The stirrer was fixed to the rack positioned 2 mm above and at a right angle to the paper.

2.6 The effects of a sealant on the CST test

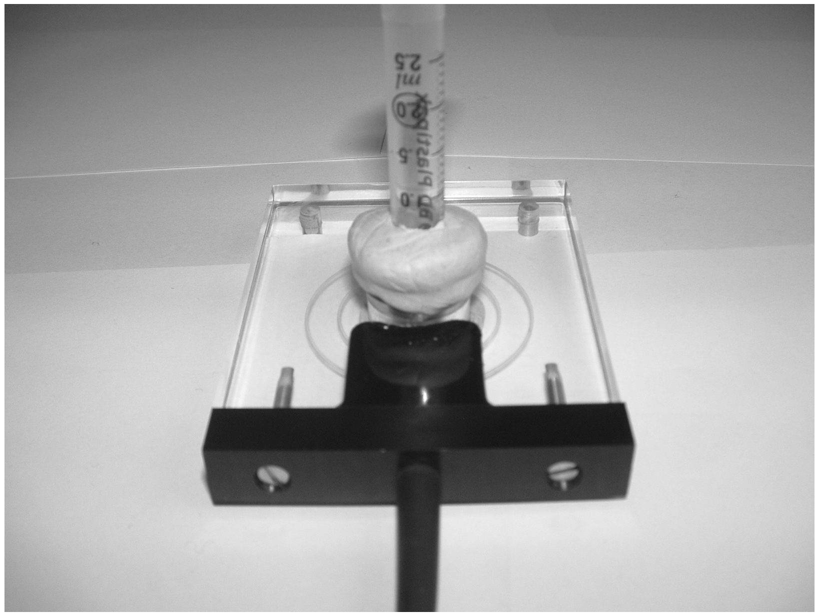
Several standard and unconventional types of sealant materials were tested in a small pilot study, including O-rings, elastic silicon, adhesive bonding material, and rubber latex. O-rings (ELE International, Bedfordshire, England) were unsuccessfully tested. It was not possible to fit the O-rings to the bottom of the funnel. Elastic silicon (Homebase, Statford, UK) was also tested, but it was not possible to apply it uniformly to the bottom of the funnel. Nevertheless, silicon showed some promising preliminary results. Further practical research may need to be undertaken on fitting silicon more effectively. Adhesive bonding material, including Blue Tack (Bostik, Stafford, UK) was also tried. Natural rubber latex was also wrapped around the bottom edge of the funnel. The expectation was that the elastic rubber would prevent or at least reduce the leakage of water between the funnel and the filter paper.

The material finally used in this research was natural rubber latex with adhesive bonding material (Blue Tack), as it showed the most promising results in preliminary studies, was easy to fit to the funnel, and could be used repeatedly over the course of the research. Sources of rubber material (e.g., party balloons) from different supermarkets (e.g., Tesco Stores, Cheshunt, UK; Sainsbury's Supermarkets, London, UK) were tested to assess if there were any differences in their applicability. Supplementary experiments showed that different rubber materials performed similarly in the context of the CST test. The material used in this research was obtained from Sainsbury's supermarket (Sainsbury's own brand; product reference numbers 747/405 and #81808; internal supplier code: UO295; bar code 0107-9852). More detailed product information can be obtained from Sainsbury's Supermarkets Ltd, 33 Holborn, London EC1N 2HT, UK.

The sealant was attached to the bottom edge of both a circular (18 mm diameter) and a rectangular funnel (18, 18 and 20 mm in length, width and depth, respectively). Two filter papers were tested: Whatman 17 chr (Whatman Plc., Brentford, UK) and Fisher 200 chr (Fisher Scientific Ltd., Loughborough, UK). The tests were carried out using synthetic sludge.

A similar rectangular funnel was tested in a previous section alongside twelve alternative filter papers where the rectangular funnel showed a considerable improvement in the test repeatability, as shown previously by Lee (1994) and Lee and Hsu (1994a). The sensor positions were not altered, and the wetting front moved in a right angle to the paper grain lines. Quantifying the amount of the leakage was estimated using a graduated pipette fixed at the top of the circular funnel placed over the filter paper (Figure 2.6.1).

Figure 2.6.1 Graduated pipette fixed on the top of the circular funnel (18 mm) of the capillary suction time test equipment



Approximately 5 ml of synthetic sludge was poured into the funnel, and the volume of the synthetic sludge that disappeared after 1 minute was recorded. This process was repeated ten times for Whatman 17 chr and Fisher 200 chr filter papers with and without using a sealant. The difference in the volumes between the funnel with and without sealant is considered to roughly represent the filtrate escaping from the gap between the funnel and the filter paper.

2.7 The effect of sludge age on the CST test

The standard CST apparatus (Whatman 17 chr paper and circular funnel) and conditions were used compare the CST values of samples of activated, digested, and primary sludges which were of known ages. The samples were obtained from Seafield wastewater treatment plant and stored inside a controlled temperature room at 10^{o}C for 1, 2, 3, 4, 5, 6, 7, or 8 days to carry out the CST tests. Three replicate CST tests were performed on the three types of sludge each day of storage.

Bacterial densities (counts/ml) were also estimated in the sludge samples stored for 4, 5, 6, 7 and 8 days. The spread plate method was used to estimate bacterial densities in sludge samples serially diluted to 10^{-8} in distilled water. 100 µl of the diluted sludge sample was spread on nutrient agar (Atlas, 1995). The nutrient agar was prepared according to the manufacturer's instructions. Autoclaved nutrient agar was poured into sterile Petri dishes, and the dishes were placed inside incubator for 48 hrs at 37°C. The numbers of colony forming units (CFU) were then counted. A plate count was considered valid if 30 to 300 CFU were counted on each plate (Atlas 1993, 1995).

2.8 Statistical analysis

Statistical analyses of the data were performed, and the figures were drawn, using Microsoft® Excel 2007, SPSS® Version 17.0 and MINITAB® Version 15.1.

The descriptive statistics for the results of the CST and SRF tests included the medians, means, standard deviations, coefficients of variation expressed as percentages (standard deviation/mean x 100) and 95% confidence intervals.

The hypothesis tests used to analyze the results of the experiments were multi-factorial analysis of variance (ANOVA) and analysis of covariance (ANCOVA) applying the General Linear Model (GLM). Full details of the hypotheses tested, and the analyses performed, with regard to the specific designs of each experiment are presented in each chapter. The justifications for using ANOVA and ANCOVA are discussed here.

The rationale for using multi-factorial ANOVA and ANCOVA was to explore the effects of different methodological factors (independent variables) on the mean CST values estimated by use of replicated CST tests (the dependent variable). Ideally, the experimental designs should all have been balanced with completely crossed factors (i.e., all the CST tests should have been performed with an equal number of replicates at all possible combinations of all the experimental factors). This is because ANOVA and ANCOVA are theoretically most powerful for balanced experimental designs (Rutherford, 2001). In practice, balanced designs were not always possible for practical reasons. The General Linear Model was consequently applied, not only because GLM provides for covariates, but also because it generates valid results for unbalanced experimental designs with missing values (Rutherford, 2001).

The results of the CST tests varied with respect to the values of certain covariates (e.g., the concentrations of sludge). This confounded the effects of other methodological factors under investigation. When the data were analyzed using ANCOVA, by controlling for the covariates, which varied linearly with respect to the CST values, then the results could be interpreted as if the CST tests had been performed at constant values of the covariates (e.g. the same concentrations of sludge).

ANOVA and ANCOVA assume homogeneity of variance i.e., that the variances in each group of data are equal. There are various tests for homogeneity of variance (e.g., Levene's test and Bartlett's test). Levene's test was used in this investigation, because it is more robust than Bartlett's test, in that it does not depend on, and is not sensitive to, the normality of the underlying data (Rutherford, 2001)

ANOVA and ANCOVA assume that the residuals (the differences between the mean values and the observed values) are normally distributed, and have a mean value of zero. There are various tests for residual normality (e.g., the Anderson-Darling test, the Kolmogorov-Smirnov test, the Shapiro-Wilk test, the Ryan-Joiner test, and the Smirnov–Cramér–von-Mises test). The Anderson-Darling test was chosen in preference to the others in this study because it is reputed to be the most powerful statistic for detecting departures from normality, and unlike some of the other normality tests, it is not so sensitive to sample size. The Anderson-Darling test generates valid results for small sample sizes < 30 and large sample sizes > 300 (Shapiro, 1980; Scholz and Stephens, 1987).

One of the main practical issues associated with the interpretation of CST and SRF tests is the high variability of the results, including unusually high or low values, termed "outliers". Outliers were identified as standardized residuals (residual/standard deviation) exceeding ± 2.0. Various transformations were explored in an attempt to homogenize the variances and normalize the residuals associated with the highly variable results of the CST and SRF tests.

Box-Cox tests (Box and Cox, 1964) were used to identify the most appropriate data transformations. The Box-Cox test is an exploratory tool which uses a maximum likelihood method to determine the optimum transformation parameters, denoted by the symbol λ. Although the optimum estimates of λ are computed as specific decimal numbers, in practice the selected values of λ corresponded to standard transformations that were easy to interpret; i.e., squares ($\lambda = 2.0$) square roots ($\lambda = 0.5$) or natural logarithms ($\lambda = 0$). Consequently squares, square roots, and logarithms ($\log_e$) were the preferred data transformations used in this investigation.

The prescribed level of significance for the results of ANOVA and ANCOVA (i.e., the F statistics) was $\alpha = 0.01$. The value of α defined the risk of making a Type I error. A Type I error results in the rejection of a null hypothesis when the hypothesis is, in fact, true. In this investigation the significance level of $\alpha = 0.01$ implied a 1 in 100 chance of a making a Type I error, so the investigator was 99% confident that the results were valid. The more conservative 0.01 level of significance was chosen in preference to the conventionally used $\alpha = 0.05$ (implying a 1 in 20 chance of making a Type I error) to compensate for violations of the assumptions that were experienced in the course of some of the analyses.

2.9 Construction of empirical models

Three empirical models will be developed in this study. The first empirical model to be considered concerns the estimation of filterability, extracted from the results of CST tests using CST device model 319 with multiple electrodes that sense the water movement. A simple linear regression model is assumed to predict filterability from the results of a CST test (Meeten and Smeulders, 1995) using equation 2.9.1:

$$CST = \beta_0 + FZ \qquad (2.9.1)$$

where CST = the predicted mean CST; β_0 = the intercept (the predicted mean CST value when the distance[4] between the stopping and starting electrodes of the CST device is zero); F = the slope of the regression line (this is the estimate of filterability, which is assumed to be a function of SRF); Z = the distance[4] between the electrodes of the device.

The second empirical model to be considered here is the prediction of the variability in the results of CST tests in terms of the physical variables associated with the tests. To date, no models have been previously been constructed for this purpose. A multiple linear regression model is tentatively proposed as follows (equation 2.9.2):

$$CST = \beta_0 + \beta_1 TSS + \beta_2 T + \beta_3 P + \beta_4 G \qquad (2.9.2)$$

where TSS = total suspended solids concentration, T = temperature; P = a variable to define the type of filter paper; G = a variable to define the funnel geometry; and β_0, β_1, β_2, β_3 and β_4 = regression coefficients.

The third empirical model to be considered here is the prediction of the variability in the results of SRF tests in terms of the results of CST tests. There has relatively little research on this topic since Baskerville and Gale (1968) first recorded that the CST was correlated with the SRF i.e. that sludge samples with a high CST also had a high SRF. In a review of trends in capillary suction time testing research, Scholz (2005) stated that for specified suspended solids contents, "CST values usually correlate well with SRF" but only a few researchers published correlations between the results of the CST and SRF tests (Tay and Jeyaseelan, 1997a 1997b; Jill *et al.,* 2003; Ma *et al.,* 2007). Progressing from correlation to regression analysis, Christensen *et al.* (1993) modelled the relationship between CST and SRF empirically using equation 2.9.3.

$$CST = c_1 SRF \mu_j W + c_2 \mu_j \qquad (2.9.3)$$

where c_1 and c_2 = coefficients related to the CST, μ_j = sludge viscosity, and W= solid content per unit volume of the filtrate.

Christensen *et al.* (1993) used CST as the dependent (predicted) variable and SRF, sludge solid content, and sludge viscosity as the independent (predictor) variables. Similarly, Ma et al. (2007) reported a relationship between the CST and the SRF settings based on SRF theory. They reported a moderate positive correlation between log CST values and log values of the filtrate viscosity and SRF based on theoretical approach. The current research, however, focuses on developing an empirical model to predict the converse relationship, i.e., the results of the SRF test from the results of the CST test. It is suggested that a potentially viable approach might be to incorporate SRF as the dependent variable, and use several independent variables associated with the results of CST tests as predictors. A multiple linear regression model is tentatively proposed, as follows (equation 2.9.4):

$$SRF = \beta_0 + \beta_1 TSS + \beta_2 CST + \beta_3 X \qquad (2.9.4)$$

where TSS = total suspended solids concentration, CST = a variable extracted from the results of CST tests e.g. filterability; X = an independent variable associated with the properties of the sludge (e.g. viscosity) and β_0, β_1, β_2, and β_3 = regression coefficients.

The multiple linear regression models proposed above need to be calibrated and validated using experimental data. The statistical assumptions of the models also need to be tested. Multiple regression models assume linearity between the dependent and independent variables which are unlikely to exist in reality, therefore transformations (e.g. logarithms, powers, and roots) may be necessary to linearize the relationships. Following the assumptions of multiple linear regression (Chatterjee *et al.,* 2000) the proposed multiple regression models assume that the independent variables are not multi-colinear, i.e. they are not correlated with each other, which may not occur in reality, and this issue must be addressed in order to construct valid models.

The suspended solids content of the sludge (TSS) is an essential component of the proposed multiple regression models, since the results of both CST and SRF tests are known to vary with respect to the TSS content of sludge. Ives (1978) reported that, at very low TSS concentrations, the CST was similar to that of water alone, whereas at higher TSS concentrations, which produced thicker cakes i.e., deeper depositions of solids onto the paper by sedimentation, the CST test provides a more accurate indication of filterability. Subsequent research found that the estimated CST values vary with respect to the TSS concentrations, not only because of variations in sludge viscosity related to solid content, but also because solids influence the thickness of the cake and its resistance to flow (Lee and Hsu, 1994a).

An important issue regarding the formulation of empirical models to predict CST or SRF values is whether sludge viscosity should be incorporated directly as a predictor variable (e.g., as applied by Christensen *et al.,* (1993) in their model to predict CST) or whether another term, which is linked to viscosity, should be substituted for viscosity. Sludge viscosity is known to be a function of temperature (El Shafei *et al.*, 2005) implying that temperature is a potential covariate or controlling variable which might substitute for viscosity.

The existence of a mechanistic relationship between temperature and sludge viscosity implies that temperature could be substituted for sludge viscosity to model the variations in the results of CST and SRF tests using multiple regression. Non-linear relationship between temperature and sludge viscosity, which can be defined by the Arrhenius equation, could be taken into account when using multiple linear regression analysis. The relationship can be linearized since $\log_e \eta$ versus 1/T where η = viscosity and T = temperature is a straight line (El Shafei *et al.*, 2005). Alternatively, polynomial terms (e.g., $T + T^2$) could be used in regression models to approximate non-linear relationships (Chatterjee *et al.,* 2000).

There are many other factors associated with the variability in the results of CST and SRF tests in addition to solid content, filterability, sludge viscosity, temperature, and the physical components of the system, e.g., the types of filter paper and funnel geometry. Ives (1978) also included the filtration area, the net filtration pressure, and the specific resistance at the chosen net filtration pressure as factors which may control the magnitude of the CST. Guan *et al.* (2003) and Scholz (2005) highlighted the importance of different floc sizes and structures to explain the variability in the dewaterability of sludge using CST tests. Cetin and Sürücü (1989) considered the influence of variable pH on the filterability of sludge. The binding of variable amounts of water onto the filter is also important (Scholz, 2005). These factors may change from one test to another, resulting in temporal variations in the physical-chemical test conditions, which may contribute towards the high residual variability of multiple CST tests on replicated sludge samples.

The models proposed above were constructed and calibrated using simple linear regression (SLR) analysis, based on the following terms (equation 2.9.5 and equation 2.9.6):

$$Y = \beta_o + \beta_1 X \qquad (2.9.5)$$

or by multiple linear regression (MLR) analysis based on the following terms:

$$Y = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + ...\beta_n X_n \qquad (2.9.6)$$

where Y= the predicted estimate of the mean value of the dependent variable, X_1, X_2, $X_{3,....}X_n$ = the independent variables, β_0 = the intercept, β_1, β_2, $\beta_{3....}$ $\beta_{n,}$= the partial regression coefficients, and n = the number of independent variables in the model. The dependent and independent variables used to calibrate the regression equations were mainly quantitative and continuous (e.g., the results of CST or SRF tests, the temperature, and the sludge concentration). Nominal variables, with no logical numerical order, (e.g., paper type and funnel type) were also included as dummy binomial variables in MLR models, coded by use of the integers 0 and 1.

The theoretical assumptions of regression analysis are strict (Chatterjee *et al.,* 2000) and it is not always possible to construct valid models. However, every attempt was made in this investigation to ensure that the models extracted from the available data did not violate any theoretical assumptions. MLR assumes linear relationships between the dependent variable and each of the independent variables. Linearity means that the mean change in Y associated with a unit change in X is additive and constant; e.g., if X_2, X_3, ..., X_n are held constant, then a unit change in X_1 should result in a mean change of β_1 in the Y variable. When empirical values of

X and Y are not linearly related, then the additive assumption is not viable, and therefore transformations of the Y and/or X variable are necessary. Some researchers simply choose the best transformation for regression analysis by trial and error (Tabachnick and Fidell, 2007). In this study, a variety of transformations (e.g., squares, cubes, square roots, and logarithms) were applied to the dependent and independent variables, following the use of Box-Cox tests (described above in relation to ANOVA and ANCOVA). The best transformations were found, which linearized the relationships between the variables, normalized the residuals, and homogenized the variances. The independent variables in a MLR model should not be co-linear; i.e., inter-correlated with each other. Co-linearity results in changes in the values of the regression coefficients, and increases the values of the standard errors, which reduces the significance levels of the regression coefficients. The regression coefficients of co-linear independent variables may not be significant, even if they are linearly related to the dependent variable, and even when the overall regression model is statistically significant (Chatterjee *et al.,* 2000). Two or more independent variables can be combined with each other (e.g., by addition, multiplication or division) to avoid co-linearity. For this reason, in this investigation, some independent variables were combined to construct multiple regression models that excluded co-linearity. Since there were many potential independent variables to select from, optimum sub-sets of variables had to be chosen to construct the best fitting MLR models. Over-fitting the models by including too many variables was avoided. The best models, which included the least number of non co-linear independent variables, were extracted from the data, to accurately predict the dependent variables, following an objective methodology, without violating any of the strict rules and assumptions imposed by regression analysis. The stepwise procedure was applied to determine the optimum sub-sets of independent variables for inclusion in the MLR models. Different groups of potential independent variables were systematically added, one by one, to a series of MLR models, and decisions were made, using objective statistical criteria, as to whether to select or exclude each variable. The potential independent variables were selected or excluded on the basis of tolerance thresholds including the values of the coefficients of determination (R^2), the variance ratios (F) obtained by ANOVA, the results of t tests on the regression coefficients, the standard errors of the estimated values of Y, the Mallow's Cp statistics, and the degree of co-linearity between the variables, denoted by variation inflation factor (VIF) statistics. The best fitting models with the following combined attributes were chosen: the least number of independent variables, the most significant t and F statistics ($p < 0.05$), a low standard error, a high value of adjusted R^2 (≥ 0.8) and minimal co-linearity ($VIF < 5$). The adjusted R^2 statistic expresses the proportion of the variance in the Y variable, which is explained by the independent variable(s). The aim was to discover the best fitting empirical models with adjusted R^2 values as close as possible to 100%, but including the least number of independent variables. The unadjusted value of R^2 generally increases with respect to the number of independent variables, and is therefore not necessarily the best estimate of the goodness of fit. The significance of each regression coefficient (intercept and slope) was determined using a t test, where the t statistic was expressed as the value of the regression coefficient divided by the standard error. The decision rule was to reject the null hypothesis (i.e., that the coefficient was zero) and accept the alternative hypothesis (i.e., that the coefficient was not zero) if the p value of the t statistic was less than the prescribed significance level of $\alpha = 0.05$. The normality of the residuals was checked using the Anderson-Darling test, as used similarly to test for residual normality in ANOVA and ANCOVA. The optimum MLR models extracted from the stepwise regression procedures used in this investigation were believed to be the most conservative, and the best possible fits that could be obtained from the available data. Attempts were made to validate the models by comparison of their predictions against the data used to calibrate the models.

Chapter Three

The Properties of Natural and Synthetic Sludges

3.1 Introduction

The aim was to find appropriate synthetic sludge formulations that approximated the physical and chemical properties of activated sludge, waste activated sludge, and digested sludge in terms of their total suspended solid concentrations (TSS), pH, supernatant turbidity (NTU), rheology (viscosity) and dewaterability (results of CST tests). The floc size distributions of the synthetic sludges were also measured as the flocculation properties have an effect on sludge dewaterability (CST test).

3.2 Chemical and physical properties

The physical and chemical properties of activated, waste activated, and digested sludges were measured. The physical and chemical properties of synthetic sludges were also compared with those of natural sludges in terms of TSS, pH, supernatant turbidity, and CST (Figures 3.2.1 to 3.2.4). The results are presented only for the three synthetic sludge concentrations that approximated the properties of natural sludges.

Figure 3.2.1 Comparison of the results of CST tests for natural and synthetic sludges

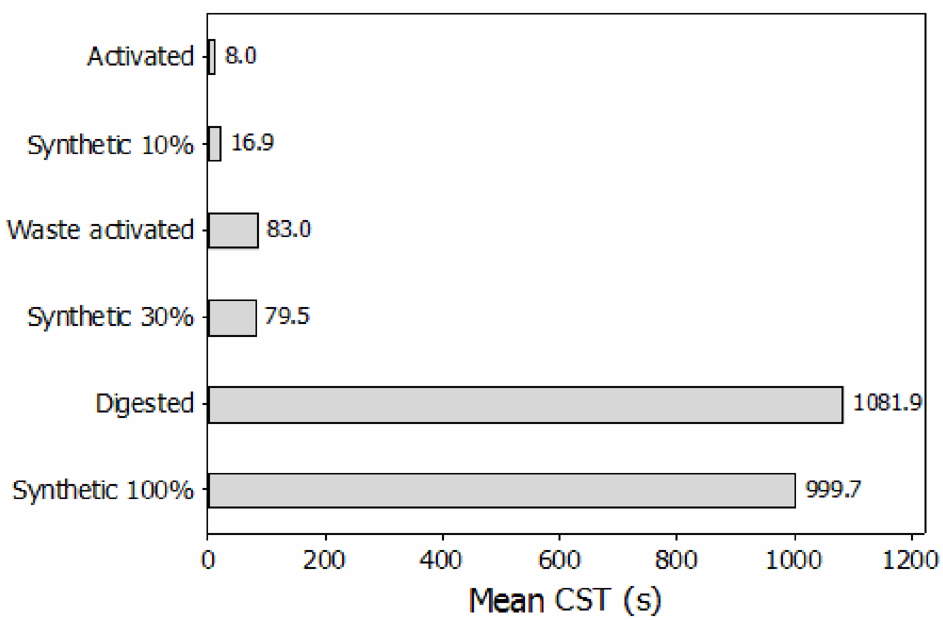


Figure 3.2.2 Comparison of the total suspended solid (TSS) concentrations of natural and synthetic sludges

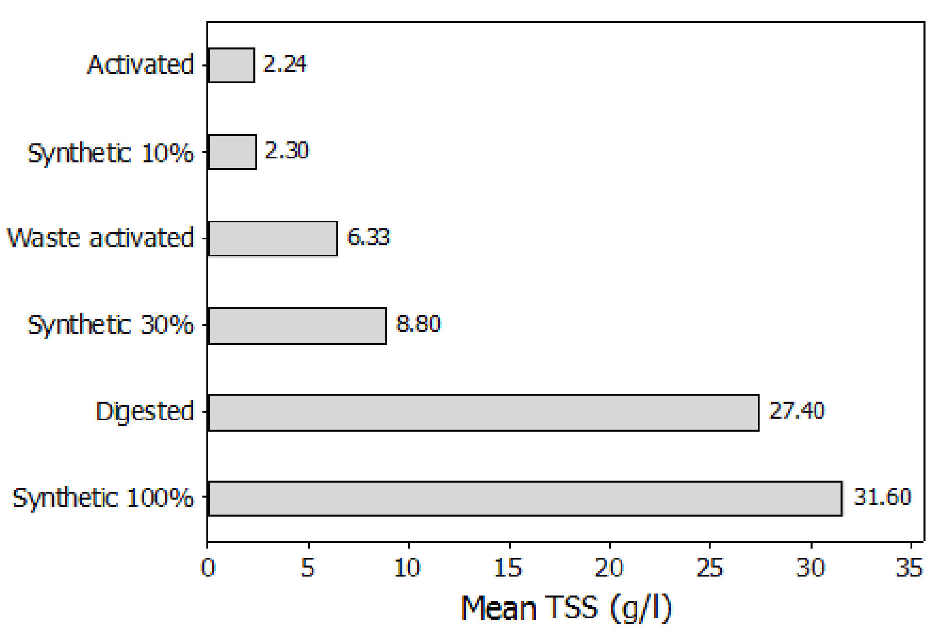


Figure 3.2.3 Comparison of the pH of natural and synthetic sludges

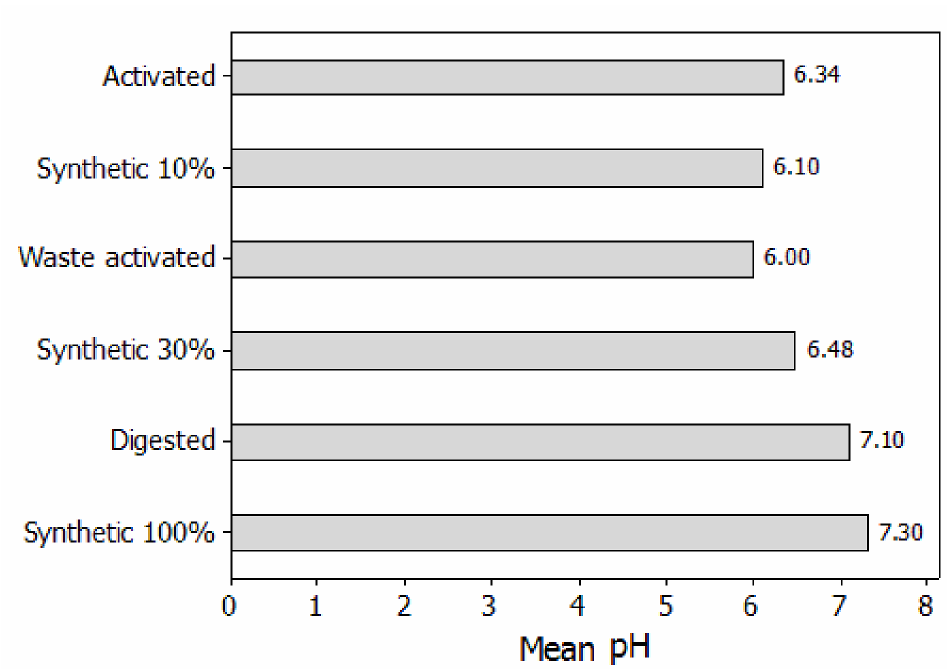
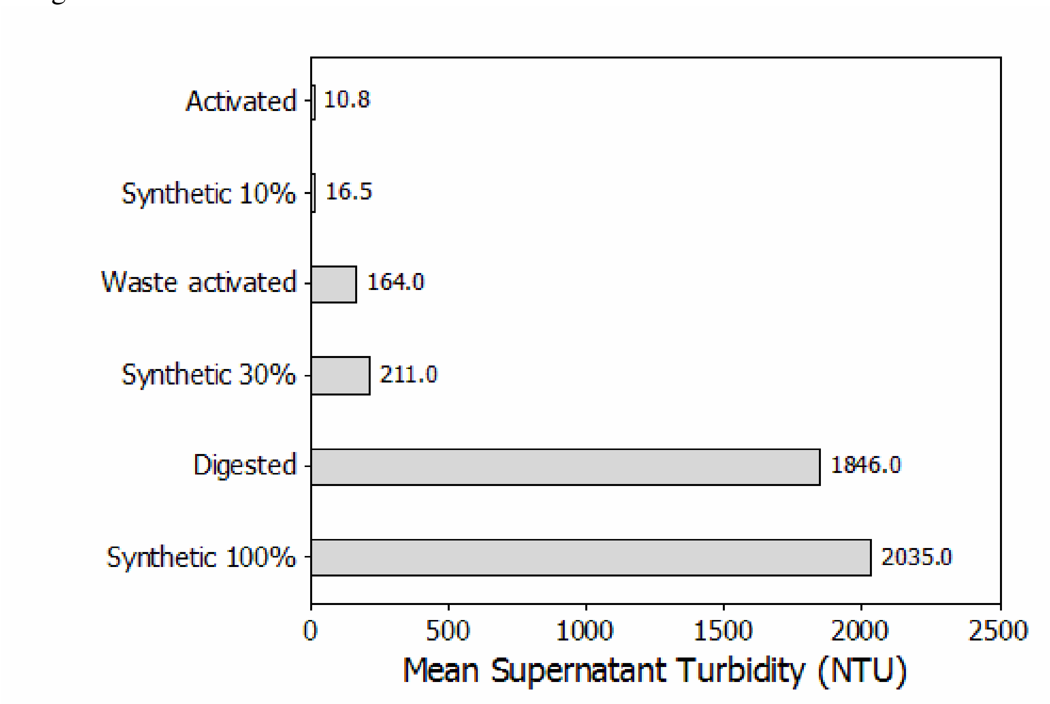


Figure 3.2.4 Comparison of the supernatant turbidity (NTU) of natural and synthetic sludges



For the synthetic sludge with 10% solid content (2.3 g/l TSS), the TSS, pH, supernatant turbidity after 30 min of settling and CST were 2.3 g/l, 6.1, 16.5 NTU and 16.9 s respectively. The corresponding values for activated sludge were 2.24 g/l, 6.34, 10.8 NTU and 8 s, respectively. The supernatant turbidity of the synthetic sludge was slightly higher than that for the activated sludge. This could be due to the small particles were in the synthetic sludge more than those in the activated sludge as indicted by the supernatant turbidity. The properties of the 10% (2.3 g/l TSS) synthetic sludge were considered to approximate activated sludge.

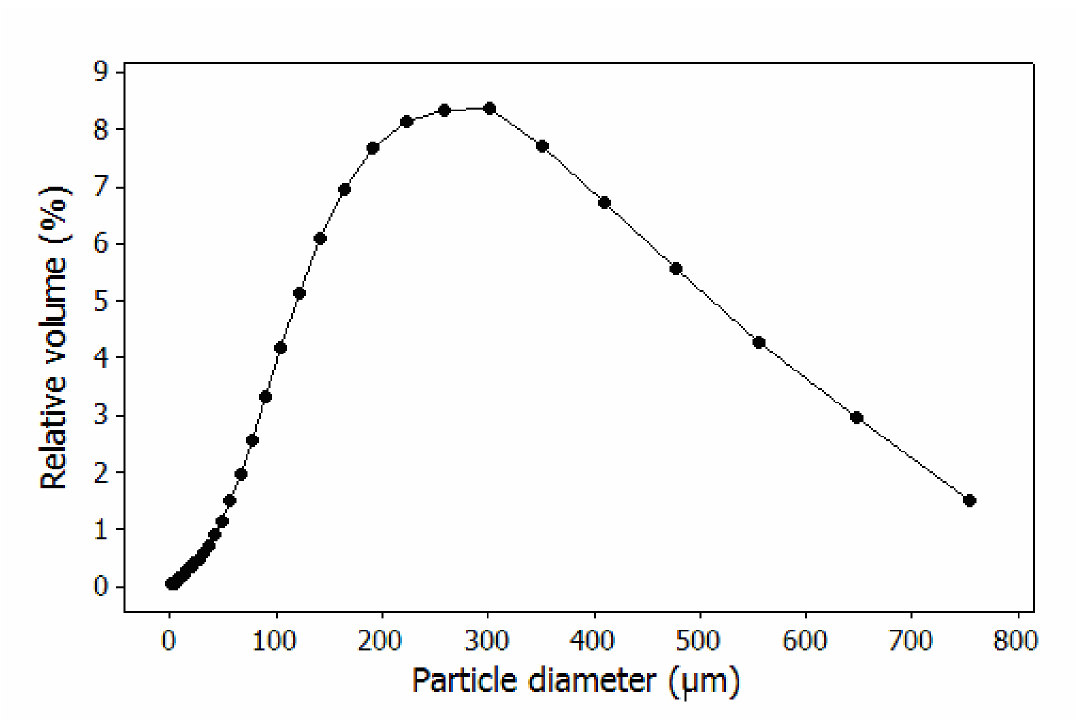
For the synthetic sludge with 30% solid content (8.80 g/l TSS), the TSS, pH, supernatant turbidity after 30 min of settling, and CST were 8.80 g/l, 6.48, 211 NTU and 79.50 s respectively. The corresponding values for activated sludge were 6.30 g/l, 6.00, 164 NTU and 83.00 s, respectively. The supernatant turbidity of the synthetic sludge was higher than that for the waste activated sludge. The CST value of the synthetic sludge (79.50 s) was very close to CST value of the waste activated sludge (83.00 s). The properties of the 30% (8.8 g/l TSS) synthetic sludge were considered to approximate waste activated sludge.

For the synthetic sludge with 100% solid content (31.60 g/l TSS) the TSS, pH, supernatant turbidity after 30 min of settling and CST were 31.60 g/l, 7.30, 2035 NTU and 999.70 s respectively, and the corresponding values for digested sludge were 27.40 g/l, 7.10, 1846 NTU and 1081.90 s, respectively. The supernatant turbidity of the synthetic sludge was higher than that for the digested sludge. The CST value of the synthetic sludge (999.70s) was close to the CST value of the digested sludge (1081.90 s). The properties of the 100% (31.6 g/l TSS) synthetic sludge were considered to approximate digested sludge.

3.3 Floc size distributions

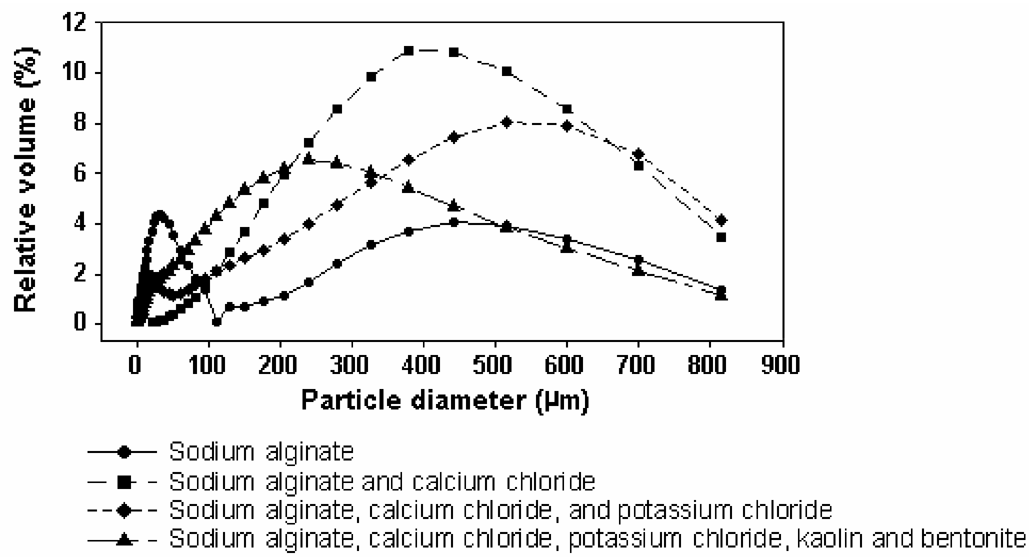
The particle size distribution of the synthetic sludge was measured using the image analyzing device Malvern Master Sizer Laser Radiation class 3B laser product (refer to section 2.1.3 for detailed description of floc size distribution measurements and specifications). The relative volumes of particle diameters in the synthetic activated sludge varied from 1 to 754 µm with a mean of 251 µm. Floc sizes of between 200 and 400 µm represented about 50% of the total volume (Figure 3.3.1).

Figure 3.3.1 Distribution of particle sizes in synthetic activated sludge



The particle size range of Sodium alginate alone was bimodal, with modes at 30 µm and 409 µm (Figure 3.3.2). The addition of Calcium chloride or Potassium chloride to Sodium alginate resulted in significant changes in the particle size distributions. The size distribution of Sodium alginate with Calcium chloride had a modal particle diameter of 409 µm (Figure 3.3.2). When Calcium chloride and Potassium chloride were added to Sodium alginate the size distribution became bimodal, with peaks of small particles 16 µm in diameter and large particles 647 µm in diameter (Figure 3.3.2). The size distribution after adding Kaolin and Bentonite clay to the suspension had a modal particle diameter of 222 µm (Figure 3.3.2)

Figure 3.3.2 Distribution of particle sizes in synthetic sludges with different ingredients



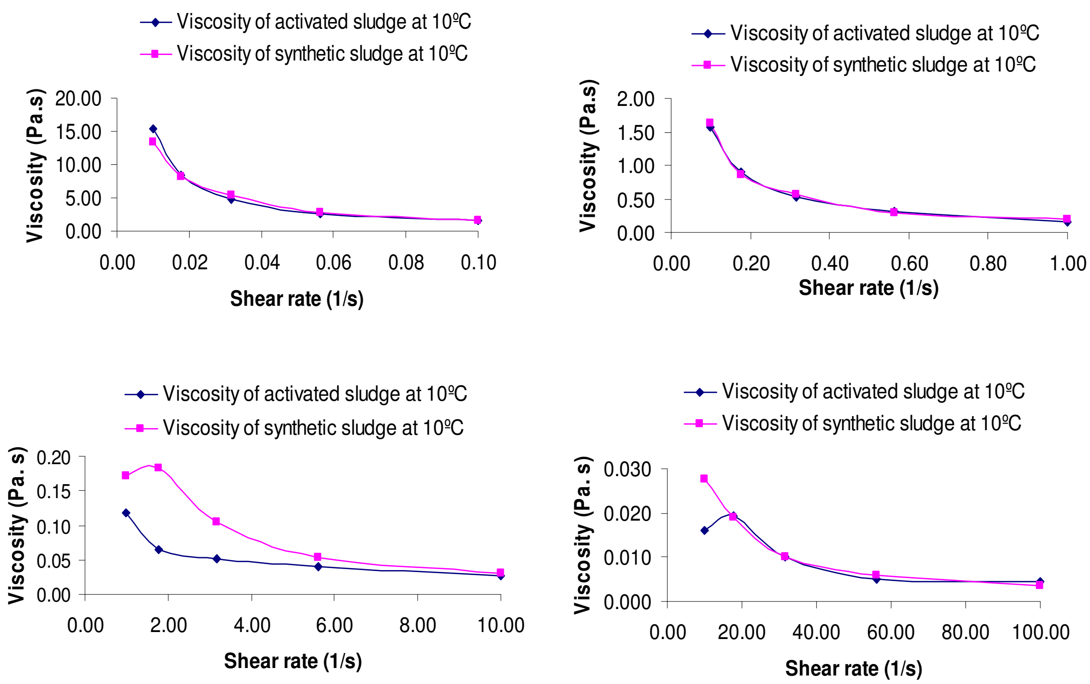
The addition of Calcium chloride and Potassium chloride created a synthetic sludge containing relatively higher frequencies of larger particles between 400µm and 700µm.

These results confirmed the existence of relationships between the ingredients, the particle size distributions, and the flocculation properties of synthetic sludges, which should be taken into account when interpreting the results of CST tests.

3.4 Viscosity

The results of investigations on the rheological properties of natural and synthetic sludges are presented in Figures 3.4.1 to 3.4.4. Figures 3.4.1a to 3.4.3b show the relationship between shear rates and viscosity for different types of sludges at different temperatures. The highest viscosities were at low shear rates, then the viscosities decreased non-linearly with respect to shear rate, reaching a plateau at about mid shear rates. The same general non-linear trend was observed for all sludges, concentrations and temperatures. For example, Figure 3.4.1a shows that the highest viscosity of activated sludge at 10 °C was 15.37 Pa.s, decreasing non-linearly to 0.000458 Pa.s from shear rates 0.01/s to 100/s. The highest viscosity of synthetic sludge with 2.3 g/l TSS concentration was 13.380 Pa.s and decreased non-linearly as described above. The viscosities of activated sludge with shear rates from 0.01/s to 100/s at temperatures of 10 ^{o}C approximated the viscosities of 10% synthetic sludge with a TSS concentration of 2.3 g/l. Similarly, the viscosity behaviour at 15 °C for both the activated and the synthetic sludges were in close agreement (Figure 3.4.1b).

Figure 3.4.1 Viscosities of activated sludge and 10% synthetic sludge
(a) 10^{o}C



(b) 15^{o}C

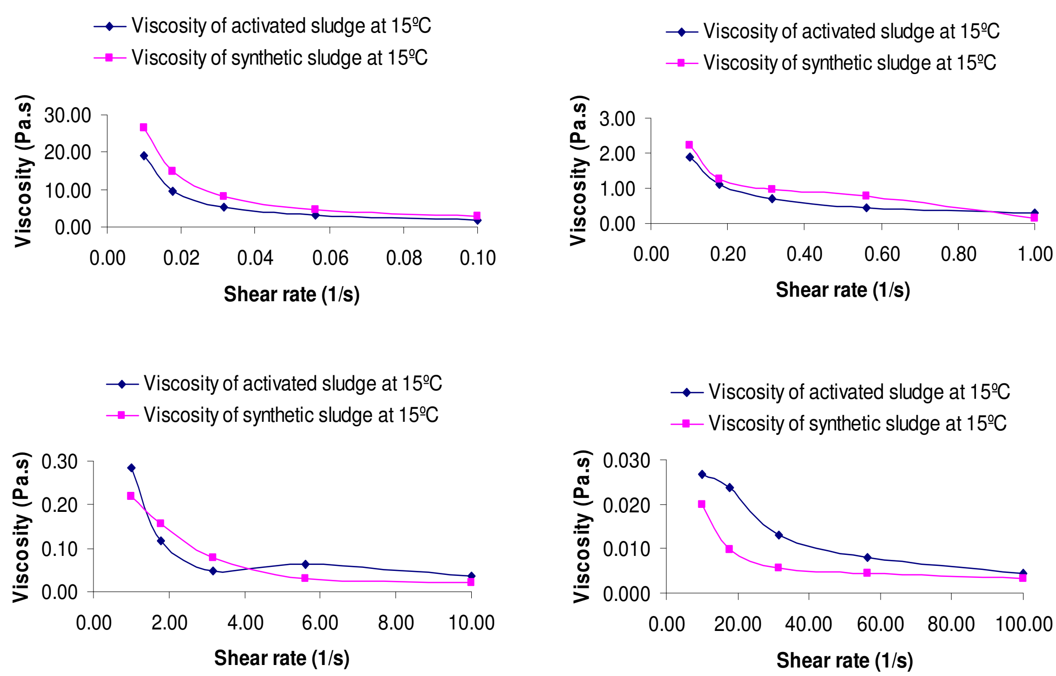
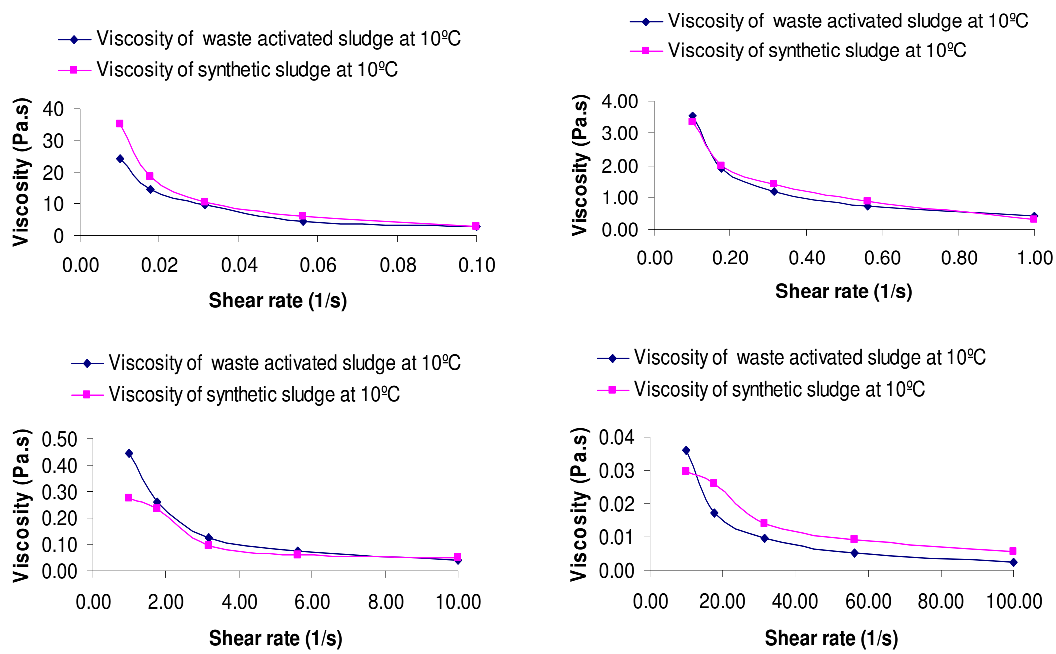


Figure 3.4.2 Viscosities of waste activated sludge and 30% synthetic sludge

(a) 10^{o}C



(b) 15^{o}C

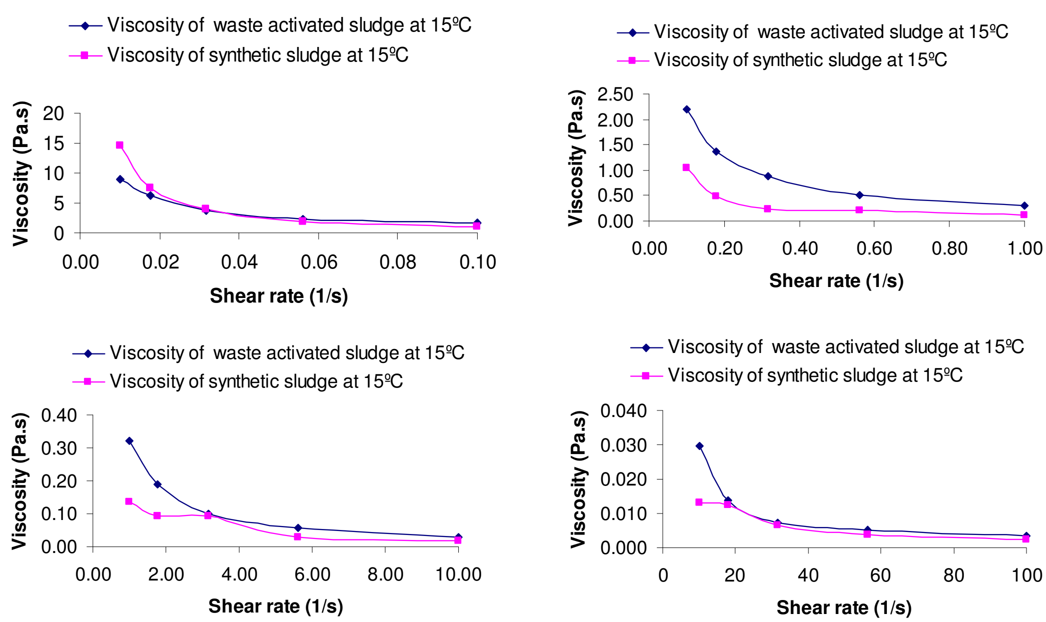
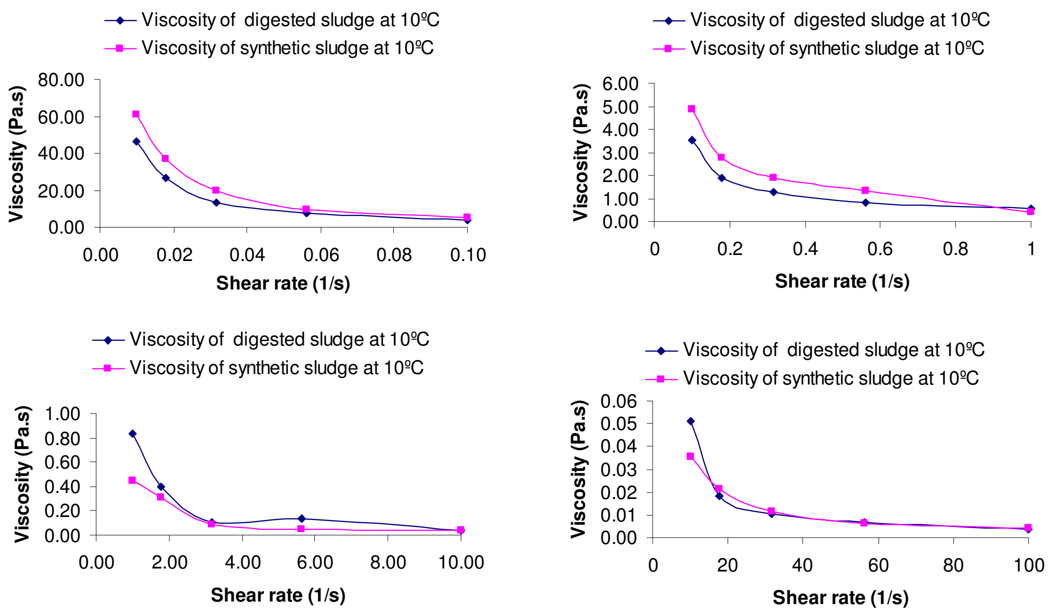
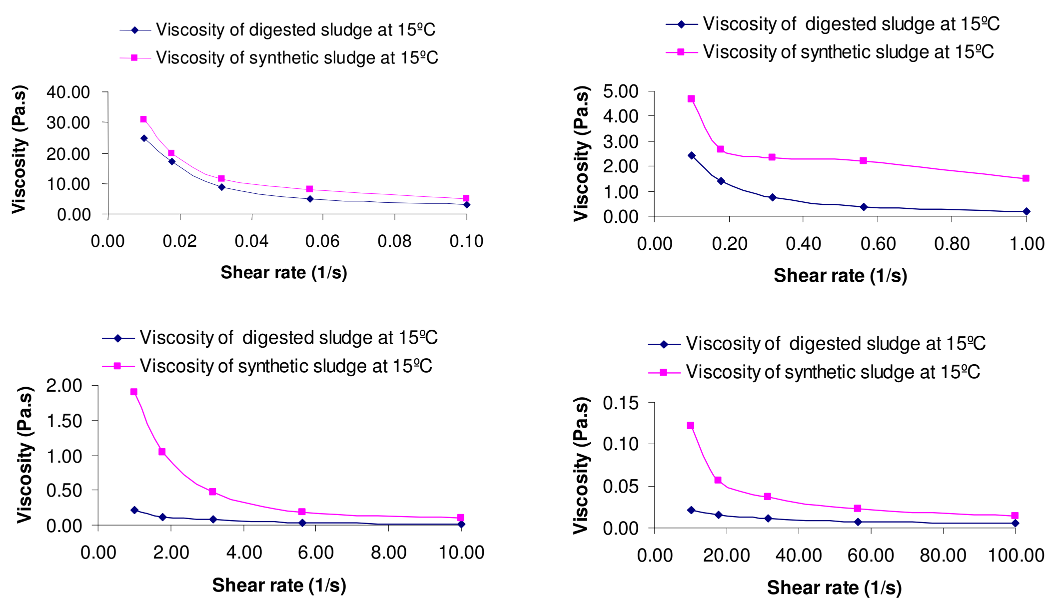


Figure 3.4.3 Viscosities of digested sludge and 100% synthetic sludge

(a) 10^{o}C



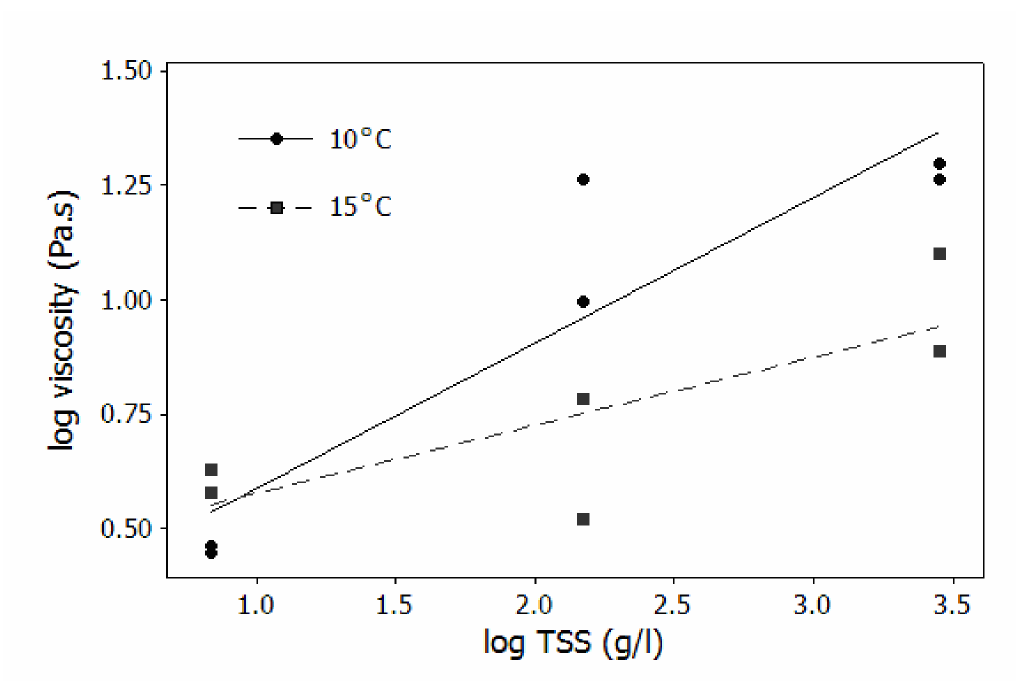
(b) 15^{o}C



The same trend was for the other two synthetic sludge concentrations (8.8 g/l and 31.6 g/l) and the waste activated and digested sludges. The viscosities of waste activated sludge with shear rates from 0.01/s to 100/s at temperatures of 10°C and 15°C approximated the viscosities of 30% synthetic sludge with a TSS concentration of 8.8 g/l at the same temperatures (Figure 3.4.2).The viscosities of digested sludge with shear rates from 0.01/s to 100/s at temperatures of 10°C and 15°C approximated the viscosities of 100% synthetic sludge with a TSS concentration of 31.6 g/l at the same temperatures (Figure 3.4.3).

The increase in viscosity with respect to an increase in sludge concentration was non-linear; however, this relationship was linearized by logarithmic transformation. At a given shear rate the log viscosity increased linearly with respect to the log TSS concentration (Figure 3.4.4).

Figure 3.4.4 Relationship between log viscosity and log synthetic sludge concentration at a constant shear rate (0.1/s)



The trend lines indicated that the rate of change in sludge viscosity with respect to TSS was higher at a temperature of 10^{o}C than 15^{o}C. Linear regression analysis indicated that the slope (the increase in viscosity per 1.0 g/l increase in TSS) at 10^{o}C was 0.316 compared to 0.148 at 15^{o}C (Table 3.4.1). An increase in TSS at 10^{o}C was responsible for a greater change in viscosity than an increase in TSS at 15^{o}C.

Table 3.4.1 Linear regression of log viscosity (Pa.s) versus log TSS (g/l) at a constant shear rate (0.1/s)

Temperature ^{o}C	Regression coefficients		t statistic	R^2 %
10	Intercept	0.273	1.72	85.1%
	Slope	0.316	4.79*	
15	Intercept	0.429	6.38	62.9%
	Slope	0.148	2.60*	

* Significant at $\alpha = 0.05$

These results confirmed the existence of significant relationships between sludge viscosity, TSS, and temperature, which should be taken into account when interpreting the results of CST tests.

3.5 The effects of sludge age on the CST test

CST tests were performed on samples of activated, digested, and primary sludges using the standard CST test apparatus and conditions (Whatman 17 chr paper and circular funnel) of different ages. The sludges were stored in an incubator and tested daily (three replicates) inside a controlled temperature room at 10^{o}C for 1, 2, 3, 4, 5, 6, 7, or 8 days. Bacterial densities (counts/ml) were estimated in the sludge samples stored for 4, 5, 6, 7 and 8 days. Linear regression analysis and ANOVA were used to test the null hypotheses that the CST estimates and the bacterial densities (dependent variables) did not vary with respect to the age of the sludges (independent variable). Box-Cox tests generated an optimum transformation parameter of $\lambda = 0$ for the CST estimates, indicating that a logarithmic ($\log_e$) transformation was justified to normalize the data prior to performing statistical analyses.

The mean CST estimates of the activated sludge samples tended to increase with age, ranging from 7.2 s to 24.1 s during the 8 day period, with erratic coefficients of variation between 2.8% and 24.7% (Table 3.5.1).

Table 3.5.1 Variations mean CST estimates with respect to the age of the sludge

Sludge	Age (days)	Mean CST (s)	Coefficient of variation (%)
Activated	1	7.2	2.8
	2	10.0	6.0
	3	14.7	3.4
	4	16.0	11.2
	5	15.2	5.7
	6	21.7	24.7
	7	24.1	8.8
	8	23.1	8.4
Digested	1	698.3	5.6
	2	659.9	12.7
	3	690.7	6.5
	4	704.8	1.9
	5	639.5	2.9
	6	603.7	5.9
	7	617.8	2.0
	8	634.0	2.9
Primary	1	68.4	10.1
	2	65.7	6.2
	3	75.0	6.6
	4	144.0	14.4
	5	97.1	14.5
	6	148.2	16.8
	7	164.9	11.2
	8	157.6	9.4

The mean CST estimates of the digested sludge tended to decline with age, ranging from 704.8s to 603.7s during the 8 day period, with erratic coefficients of variation between 1.9% and 12.7% (Table 3.5.1). The mean CST estimates of the primary sludge tended to increase with age, ranging from 65.7 s to 164.9 s during the 8 day period, with erratic coefficients of variation between 6.2% and 16.8% (Table 3.5.1). There was no evidence to indicate that the coefficients of variation increased or decreased systematically with respect to the ages of the sludges.

The logarithmically transformed estimates of the CST were regressed on the ages of the sludges. The fitted regression lines are displayed in Figure 3.5.1 the null hypotheses of regression analysis were rejected at $\alpha = 0.01$. The intercepts and slopes were significantly different from zero at the 0.01 level, and the regressions explained significant proportions of the variability in the dependent variable (Table 3.5.2). The p values of the Anderson-Darling tests revealed that the residuals did not deviate significantly from normality (Table 3.5.2).

The linear regression equations indicated that when testing activated sludge the log CST increased by 0.162 for every day of storage. For digested sludge the log CST decreased by -0.017 for every day of storage. For primary sludge the log CST increased by 0.143 for every day of storage.

Figure 3.5.1 Regression of log CST(s) on age (days) for activated, digested, and primary sludges

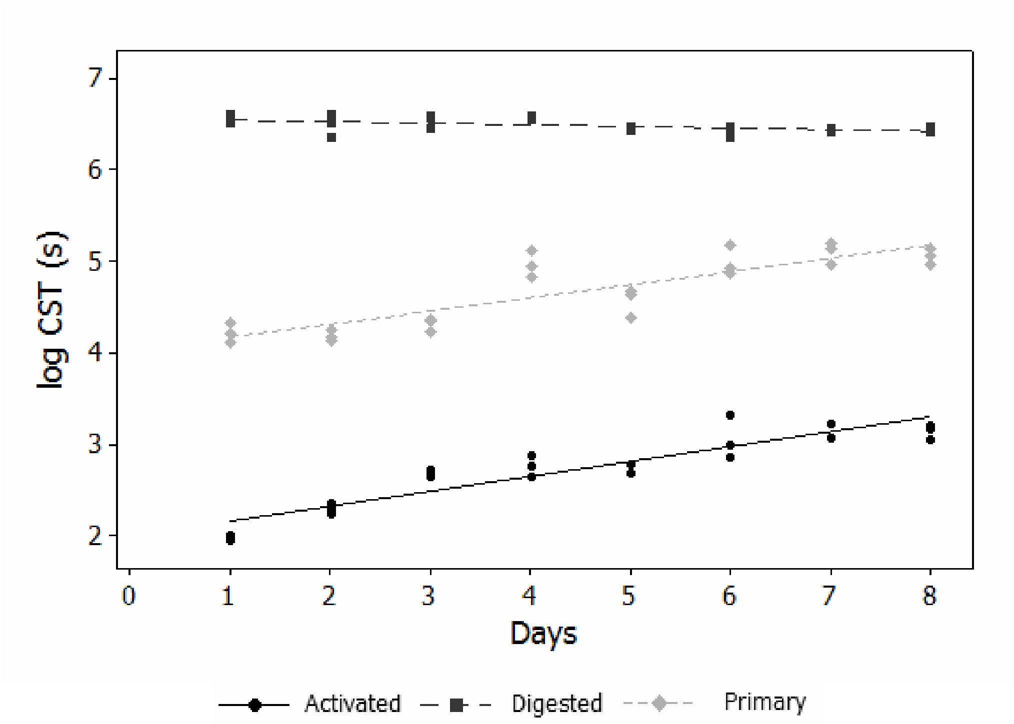


Table 3.5.2 Linear regression of log CST (s) versus sludge age (days)

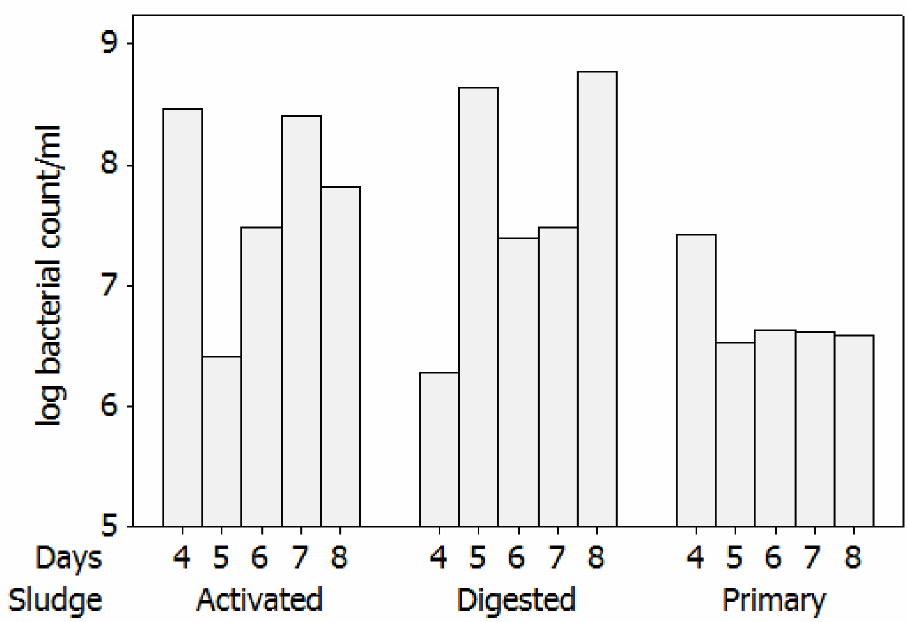
Sludge	Regression coefficients		t statistic	P	F statistic	p	R^2 (%)	Residual normality P
Activated	Intercept	1.998	27.28	0.000*	125.12	0.000*	85.0%	0.673
	Slope	0.162	11.19	0.000*				
Digested	Intercept	6.562	223.49	0.000*	9.10	0.006*	29.3%	0.199
	Slope	-0.017	-3.02	0.006*				
Primary	Intercept	4.028	43.95	0.000*	62.56	0.000*	74.0%	0.161
	Slope	0.143	7.91	0.000*				

* Significant at $\alpha = 0.01$

3.6 The bacterial densities in natural sludges

The bacterial densities varied with respect to the ages of the three types of sludge (Figure 3.6.1).

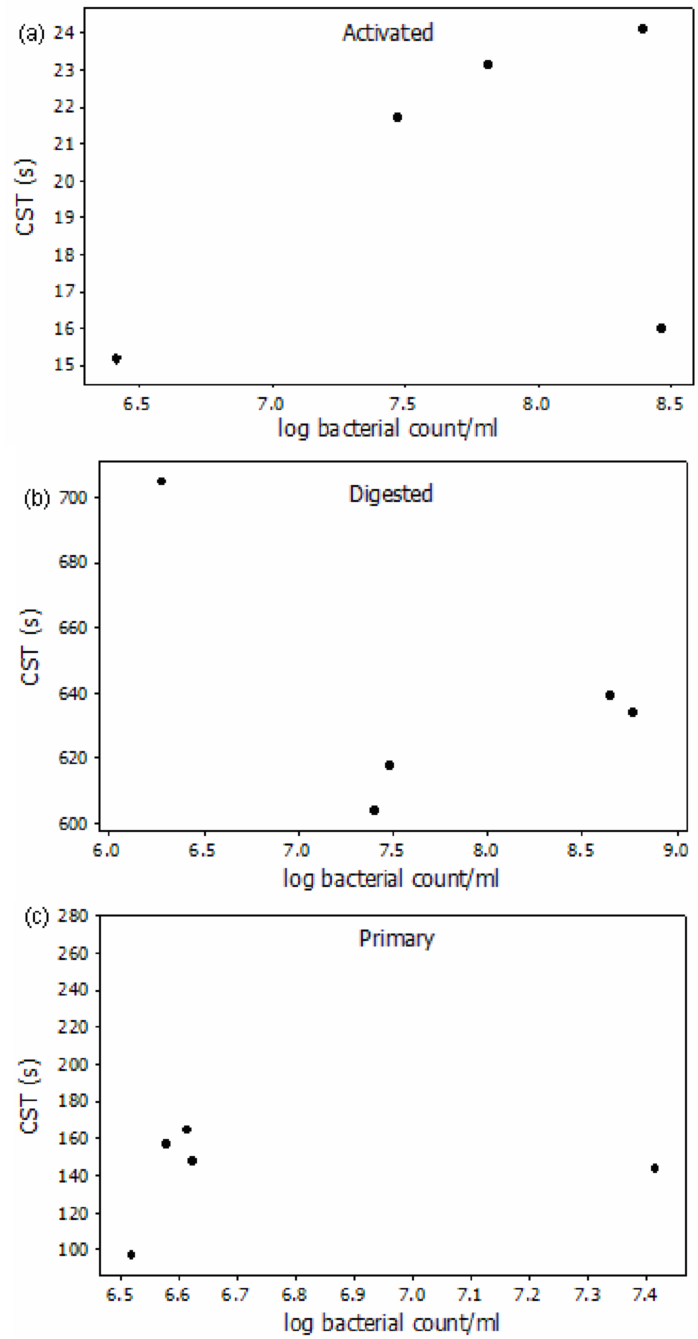
Figure 3.6.1 Variations in log bacterial counts/ml with respect to the age (days) of activated, digested, and primary sludges



The mean CST estimates for the three sludges were plotted against the bacterial densities, and non-linear trends were observed (Figure 3.6.2).

It is concluded that the CST estimates for the activated and primary sludge samples tended to increase with respect to bacterial density, whereas the CST estimates for the digested sludge samples tended to decrease with respect to bacterial density.

Figure 3.6.2 Relationships between the CST (s) and the log bacterial counts/ml in (a) activated, (b) digested, and (c) primary sludge



3.7 Discussion

The most important outcome was that synthetic sludges were successfully developed with generally similar properties to natural sludges. In terms of CST, pH and supernatant turbidity, 10% (2.3 g/l TSS) synthetic sludge approximated the properties of activated sludge; 30% (8.8 g/l TSS) synthetic sludge approximated the properties of waste activated sludge; and 100% (31.6 g/l TSS) synthetic sludge approximated the properties of digested sludge.

When natural (primary, activated, and digested) sludges were stored for 1 to 8 days, the rates of change in the dewatering properties of the sludges with respect to time were not constant, and varied with respect to the densities of bacteria. It is therefore not feasible to formulate a single time correction factor which might be used in practice to correct for the bias in the results of CST tests associated with the storage of natural sludge samples over a period of time. The development of the synthetic sludges was therefore crucial because it permitted experimental studies to be performed on the effects of modifying the conditions and the methodologies of CST tests using synthetic sludge samples with known physical and chemical properties that were stable over time because they were not subject to microbial activity.

The viscosities of both synthetic and natural sludges at 10 ºC and 15 ºC decreased with respect to an increase in shear rate. The viscosity declined as a result of the break-up of the flocs which occurs at higher shear rates (Örmeci and Vesilind, 2000). The change in viscosity with respect to shear rate became less as the shear rate increased, so the viscosity tended towards a constant at higher shear rates. When the viscosity approaches linearity at higher shear rates, this is called the limit viscosity, interpreted as being the viscosity of the sludge corresponding to the maximum dispersion of the flocs under the influence of a high shear rate (Tixier et al., 2003). Consequently the viscosity of the synthetic sludge (with different solid concentrations), activated, waste activated, and digested sludges were very similar when they contained similar TSS concentrations.

This investigation confirmed that TSS concentration is a major factor influencing the rheological properties of sludge (Dentel and Abu-Orf, 1995; Tixier et al., 2003). An exponential relationship exists between TSS and viscosity (El Shafei *et al.* 2005), since the interactions between the suspended particles depend on their density, which in turn influences fluidity. This investigation also confirmed that as the solid content increases, the sludge suspensions develop non-Newtonian characteristics. The viscosity of the sludge samples was typically a non-linear function of their shear rate. The effect of temperature on the viscosity varied with respect to the solid content of the sludge. It is therefore virtually impossible to predict the viscosity of a sludge sample, given only its suspended solids content and the temperature (Dentel, 1997; Forster, 2002; Tixier *et al*., 2003; Hasar *et al*., 2004). Viscosity is also sensitive to the density of filamentous bacteria, which are associated with a large hysteresis area, so that the bacteria increase the viscosity even at low suspended solid concentrations (Tixier *et al*., 2003). Therefore, cellulose fibres were used in the synthetic sludge to simulate filamentous bacteria common in natural sludge.

Synthetic sludges with different ingredients exhibited different particle size distributions. An elevated frequency of larger floc particles 400µm to 700 µm in diameter was associated with the addition of Calcium chloride and Potassium chloride to the mixtures. These results confirmed the influence of cations on the flocculation behaviour of Sodium alginate, Kaolin, and Bentonite particles in synthetic sludges (Nguyen *et al.* 2006; 2007; 2008).

Jin *et al* (2003) reported that the mean floc sizes in samples of activated sludge ranged from 55 µm to 311 µm. The mean particle size of 251 µm observed in the synthetic activated sludge formulated in this study was therefore within the expected range for natural sludge; however, the floc sizes of synthetic sludges and natural sludges are not directly comparable, since the particle size distributions of natural sludges depend on the treatment plant operating conditions, including the flow rate and stirring rate. For example, Chaignon *et al.,* (2002) reported that the modal floc particle diameter in a sample of activated sludge was about 200 µm, i.e. slightly smaller than that of the synthetic activated sludge used in this study; however, when the stirring rate was increased, the flocs broke up into smaller sized particles, of about 70µm in diameter.

The overall conclusion of the preliminary investigations on the properties of natural and synthetic sludges was that in order to interpret the results of CST tests, a multitude of interacting sludge-specific factors, including the age, the temperature, the viscosity, the particle size distribution, the presence of cations, and the flocculation properties should be taken into account. Variations in these characteristics from one sample to another may be considered to be integral causes of the wide variability observed in the results of CST tests.

Chapter Four

Testing the effects of different CST testing components on the CST results♣

4.1 The effects of the funnel geometry

4.1.1 Introduction

The aim was to explore the effects of different types of funnel geometry on the results of replicated CST tests using natural and synthetic sludges with different types of filter paper.

4.1.2 Experimental design

The experimental design was multi-factorial, incorporating funnel geometry, sludge type and filter paper type. The effects of three funnel geometries (a circular 10.0 mm funnel; a circular 18.0 mm funnel and a rectangular funnel, with a volume equivalent to an 18.0 mm circular funnel) were explored. Gully pot, primary, surplus activated, and synthetic sludge (Table 4.1.1) were tested with 13 types of filter paper

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♣ The content of this chapter has been published in the following journal papers:

Sawalha, O., and Scholz, M. 2007. Assessment of capillary suction time (CST) test methodologies. *Environmental Technology,* 28, 1377-1386.

Sawalha, O., and Scholz, M. 2009. Innovative enhancement of the design and precision of the capillary suction time testing device. *Water Environment Research,* 81, 2344-2352

(Table 4.1.2). All the tests were performed at 20 ±1°C and not stirred, approximating standard CST test conditions.

Table 4.1.1 Properties of the natural sludges used in the experiments

Sludge type	Sludge properties		
	Total Suspended Solids TSS (g/l)	pH	Turbidity (NTU)
Gully pot 1	411.65	6.0	+
Gully pot 2	423.63	7.5	+
Primary 1	28.25	6.4	+
Primary 2	33.50	6.5	+
Surplus activated 1	2.34	7.0	1798
Surplus activated 2	8.34	7.2	1365

+ values exceeded the upper limit of turbidity measured by the device

Table 4.1.2 Types of filter papers used in the experiments

Filter paper
B440 [a]
BF3 [a]
BF4 [a]
Carlson EE10H [i]
Fisher 200 chr [a]
FN30 [a]
FN8 [a]
HOVBO TO w/s [i]
MN280 [a]
S1107 [i]
SS 3205 [i]
SS 3324 chr [a]
Whatman 17 chr [a]

[a] anisotropic; [i] isotropic

To optimize the power of statistical analysis there should ideally be an equal number of replicates for each factor combination; however, the number of replicates in each cell of the factorial design matrix (Table 4.1.3) varied from 5 to 30 and not every possible factor combination was used, i.e., the three types of funnel were not tested against all available types of filter papers and sludges. Consequently a three-way balanced ANOVA design was not justified. Four separate two-way ANOVAs, to test for the main effects (filter paper and funnel geometry) with interactions (filter paper x funnel geometry) on the mean CST estimates for each of the four sludge types were conducted. Logarithmic transformations were performed, following the results of Box-Cox tests, to normalize the skewed distributions; however, due to violations of the assumptions of ANOVA, post-hoc multiple comparison tests were compromised. One-way Kruskal-Wallis tests were performed as a non-parametric alternative.

Table 4.1.3 Factorial design matrix to investigate the effects of funnel geometry

Funnel	Paper	Sludge			
		Surplus activated	Synthetic	Primary	Gully pot
10 mm circular	B440	20	5	0	0
	BF3	20	5	0	0
	BF4	20	5	0	0
	Carlson EE1OH	5	0	0	20
	Fisher 200 chr	25	5	0	20
	FN30	20	5	0	0
	FN8	0	5	0	0
	HOVBO TO w/s	5	0	0	20
	MN280	20	0	0	0
	SS 1107	5	0	0	20
	SS 3205	5	0	0	20
	SS 3324 chr	5	0	0	20
	Whatman 17 chr	25	5	0	20
18 mm circular	B440	0	0	6	0
	BF3	0	0	6	0
	BF4	0	0	6	0
	Carlson EE1OH	0	5	5	30
	Fisher 200 chr	0	5	11	30
	FN30	0	0	6	0
	FN8	0	0	6	0
	HOVBO TO w/s	0	5	5	30
	SS 1107	0	5	5	30
	SS 3205	0	5	5	30
	SS 3324 chr	0	5	5	30
	Whatman 17 chr	0	5	11	30
Rectangular	B440	20	5	6	0
	BF3	20	5	6	0
	BF4	20	5	6	0
	Carlson EE1OH	5	0	5	20
	Fisher 200 chr	25	5	11	20
	FN30	20	5	6	0
	FN8	0	5	6	0
	HOVBO TO w/s	5	0	5	20
	MN280	20	0	0	0
	SS 1107	5	0	5	20
	SS 3205	5	0	5	20
	SS_3324_chr	5	0	5	20
	Whatman 17 chr	25	5	11	20

4.1.3 Results

The descriptive statistics for the CST tests to investigate the effects of funnel geometry are presented in Table 4.1.4.

Table 4.1.4 Descriptive statistics for CST the tests (effects of funnel geometry)

Funnel	Paper	Min	Max	Median	Mean	SD	CV%
			Surplus activated sludge				
10 mm circular	B440	11.2	19.7	15.8	15.8	2.2	13.7
Rectangular	B440	3.0	3.8	3.4	3.4	0.2	6.7
10 mm circular	BF3	17.4	21.8	20.1	19.6	1.3	6.8
Rectangular	BF3	6.4	8.7	7.5	7.5	0.7	9.4
10 mm circular	BF4	16.5	28.7	23.6	23.9	3.0	12.5
Rectangular	BF4	3.2	4.0	3.5	3.6	0.2	7.0
10 mm circular	Carlson EE1OH	22.5	36.4	27.1	28.8	5.3	18.2
Rectangular	Carlson EE1OH	9.2	15.4	12.3	12.6	2.6	20.7
10 mm circular	Fisher 200 chr	9.5	25.9	12.6	14.6	5.0	34.1
Rectangular	Fisher 200 chr	3.4	5.4	4.1	4.1	0.5	12.7
10 mm circular	FN30	9.8	13.9	12.5	12.3	1.3	10.4
Rectangular	FN30	2.3	3.2	2.7	2.7	0.2	8.8
10 mm circular	HOVBO TO w/s	6.3	19.2	7.3	11.5	6.6	57.6
Rectangular	HOVBO TO w/s	4.0	4.8	4.3	4.3	0.3	6.8
10 mm circular	MN280	10.1	14.2	12.8	12.6	1.1	9.0
Rectangular	MN280	4.0	5.9	5.0	4.9	0.5	10.1
10 mm circular	SS 1107	20.9	34.2	30.1	28.0	6.2	22.1
Rectangular	SS 1107	14.5	16.2	15.0	15.3	0.8	5.0
10 mm circular	SS 3205	18.6	34.0	21.4	23.6	6.0	25.4
Rectangular	SS 3205	3.2	5.0	3.8	4.0	0.8	20.0
10 mm circular	SS 3324 chr	7.8	16.7	13.5	12.8	3.5	27.6
Rectangular	SS 3324 chr	3.2	3.7	3.5	3.5	0.2	5.3
10 mm circular	Whatman17 chr	12.3	28.2	16.0	16.8	3.2	19.2
Rectangular	Whatman17 chr	4.6	7.5	5.7	5.9	0.7	11.8
			Synthetic sludge				
10 mm circular	B440	211.0	332.0	281.6	274.9	44.3	16.1
Rectangular	B440	16.6	21.9	20.1	20.0	2.1	10.5
10 mm circular	BF3	107.0	126.6	124.6	119.7	8.5	7.1
Rectangular	BF3	21.4	23.6	22.3	22.4	0.8	3.6
10 mm circular	BF4	182.1	265.0	203.2	214.4	32.0	14.9
Rectangular	BF4	26.8	31.4	29.4	28.9	1.9	6.4
18 mm circular	Carlson EE1OH	48.1	56.6	56.2	54.6	3.6	6.7
10 mm circular	Fisher 200 chr	94.4	132.5	111.3	112.4	13.9	12.4
18 mm circular	Fisher 200 chr	20.5	23.6	21.4	21.7	1.2	5.4
Rectangular	Fisher 200 chr	16.9	18.3	17.2	17.5	0.6	3.3
10 mm circular	FN30	104.5	123.1	119.8	116.9	7.4	6.3
Rectangular	FN30	13.8	18.3	15.2	15.7	1.8	11.6
10 mm circular	FN8	57.2	70.9	68.5	65.8	5.8	8.8
Rectangular	FN8	12.5	14.3	12.7	13.0	0.7	5.7
18 mm circular	HOVBO TO w/s	29.3	32.1	30.2	30.5	1.1	3.5
18 mm circular	SS 1107	28.3	37.6	29.5	31.1	3.9	12.4
18 mm circular	SS 3205	42.6	47.9	46.6	46.0	2.1	4.5

Table 4.1.4 continued

Funnel	Paper	Min	Max	Median	Mean	SD	CV%
18 mm circular	SS 3324 chr	36.0	40.5	39.0	38.2	1.9	5.0
10 mm circular	Whatman 17 chr	47.0	91.4	87.7	78.3	18.3	23.4
18 mm circular	Whatman 17 chr	14.0	19.4	18.5	17.2	2.4	14.2
Rectangular	Whatman 17 chr	11.4	13.1	12.8	12.5	0.7	5.5
			Primary sludge				
18 mm circular	B440	14.4	23.4	16.2	17.3	3.3	19.4
Rectangular	B440	9.6	12.4	10.7	10.8	1.0	9.6
18 mm circular	BF3	54.2	152.1	65.6	77.5	36.9	47.7
Rectangular	BF3	23.7	36.4	33.0	32.4	4.7	14.6
18 mm circular	BF4	19.4	35.1	21.9	23.7	5.9	24.8
Rectangular	BF4	11.7	13.8	13.0	13.0	0.7	5.4
18 mm circular	Carlson EE1OH	82.1	234.3	90.0	122.6	64.7	52.8
Rectangular	Carlson EE1OH	79.9	103.9	90.3	91.5	9.6	10.4
18 mm circular	Fisher 200 chr	15.0	72.1	19.7	38.4	24.9	64.9
Rectangular	Fisher 200 chr	10.4	36.7	15.6	22.0	11.4	51.8
18 mm circular	FN30	10.8	19.1	12.8	14.4	3.6	25.4
Rectangular	FN30	7.4	9.9	8.3	8.6	1.0	11.5
18 mm circular	FN8	20.9	41.9	31.8	31.1	8.7	27.9
Rectangular	FN8	13.7	27.6	17.8	19.3	5.1	26.6
18 mm circular	HOVBO TO w/s	32.2	93.2	57.6	66.1	25.5	38.6
Rectangular	HOVBO TO w/s	11.7	13.4	12.3	12.6	0.7	5.8
18 mm circular	SS 1107	14.9	43.9	29.8	28.5	10.8	38.1
Rectangular	SS 1107	14.0	22.8	14.6	16.7	3.7	22.3
18 mm circular	SS 3205	11.9	44.5	18.6	22.0	13.4	60.7
Rectangular	SS 3205	9.8	13.4	12.0	11.6	1.6	13.5
18 mm circular	SS 3324 chr	22.4	90.2	38.4	49.1	28.2	57.3
Rectangular	SS 3324 chr	29.1	32.0	30.8	30.5	1.1	3.6
18 mm circular	Whatman 17 chr	36.4	118.0	53.0	58.9	24.1	40.9
Rectangular	Whatman 17 chr	19.5	45.6	35.8	34.9	8.8	25.3
				Gully pot sludge			
10 mm circular	Carlson EE1OH	309.8	1009.3	553.9	577.8	210.7	36.5
18 mm circular	Carlson EE1OH	78.0	227.2	135.1	143.4	47.1	32.9
Rectangular	Carlson EE1OH	67.4	149.0	96.9	99.6	24.8	24.9
10 mm circular	Fisher 200 chr	112.8	309.0	210.7	207.2	56.6	27.3
18 mm circular	Fisher 200 chr	44.2	105.1	66.4	67.3	13.9	20.6
Rectangular	Fisher 200 chr	31.9	65.8	39.9	41.7	8.9	21.3
10 mm circular	HOVBO TO w/s	60.6	606.5	110.3	191.9	187.4	97.7
18 mm circular	HOVBO TO w/s	20.9	204.6	41.1	67.3	53.6	79.7
Rectangular	HOVBO TO w/s	11.3	96.4	25.4	40.3	27.6	68.5
10 mm circular	SS 1107	88.8	246.0	146.0	149.5	38.2	25.6
18 mm circular	SS 1107	39.3	80.2	53.9	53.3	10.0	18.7
Rectangular	SS 1107	26.6	49.4	35.7	36.0	5.9	16.4
10 mm circular	SS 3205	88.0	202.5	125.9	129.2	28.5	22.0
18 mm circular	SS 3205	25.6	112.7	51.7	53.4	17.6	32.9
Rectangular	SS 3205	30.0	85.6	50.6	51.1	14.2	27.7
10 mm circular	SS 3324 chr	158.5	341.3	242.1	231.4	53.1	23.0
18 mm circular	SS 3324 chr	45.0	105.2	65.7	71.4	17.5	24.6
Rectangular	SS 3324 chr	32.1	64.4	51.2	50.9	8.3	16.3
10 mm circular	Whatman 17 chr	166.6	386.3	250.5	254.2	65.0	25.6
18 mm circular	Whatman 17 chr	52.7	105.7	82.5	80.9	13.5	16.7
Rectangular	Whatman 17 chr	37.3	70.0	56.3	55.5	8.8	15.9

Min, Minimum; Max, Maximum; SD, Standard Deviation; CV%, Coefficient of Variation

The null hypotheses of ANOVA that the funnel geometries and the filter papers had no effects on the mean CST estimates were rejected for the four types of sludge, indicated by p values < 0.01 for the F statistics (Table 4.1.5). Significant interactions between the filter paper types and the funnel geometries (denoted Funnel x Paper in Table 4.1.5) were also significant at p < 0.01 for surplus activated, synthetic, and gully plot sludge.

Table 4.1.5 Two-way ANOVA to compare the mean CST estimates with respect to funnel geometry and filter paper type

Sludge	Source of variance	Type III Sum of Squares	Degrees of Freedom	Mean Square	F statistic	p
Surplus activated	Funnel	14.675	1	14.675	3232.484	0.000*
	Paper	5.610	11	0.510	112.339	0.000*
	Funnel x Paper	2.014	11	0.183	40.336	0.000*
	Error	1.480	326	0.005		
	Total	342.559	350			
Synthetic	Funnel	12.962	2	6.481	2939.279	0.000*
	Paper	2.610	11	0.237	107.617	0.000*
	Funnel x Paper	0.316	7	0.045	20.462	0.000*
	Error	0.185	84	0.002		
	Total	290.323	105			
Primary	Funnel	2.254	1	2.254	89.883	0.000*
	Paper	9.349	11	0.850	33.888	0.000*
	Funnel x Paper	0.645	11	0.059	2.339	0.012
	Error	3.260	130	0.025		
	Total	327.513	154			
Gully pot	Funnel	30.835	2	15.417	656.710	0.000*
	Paper	11.763	6	1.961	83.510	0.000*
	Funnel x Paper	0.782	12	.065	2.776	0.001*
	Error	11.011	469	.023		
	Total	1875.412	490			

* Significant at $\alpha = 0.01$

The Anderson-Darling tests (Table 4.1.6) indicated that the residuals deviated strongly from normality at p < 0.01, and the p values < 0.01 for Levene's test indicated that the variances were not equal across all combinations of factors (Table

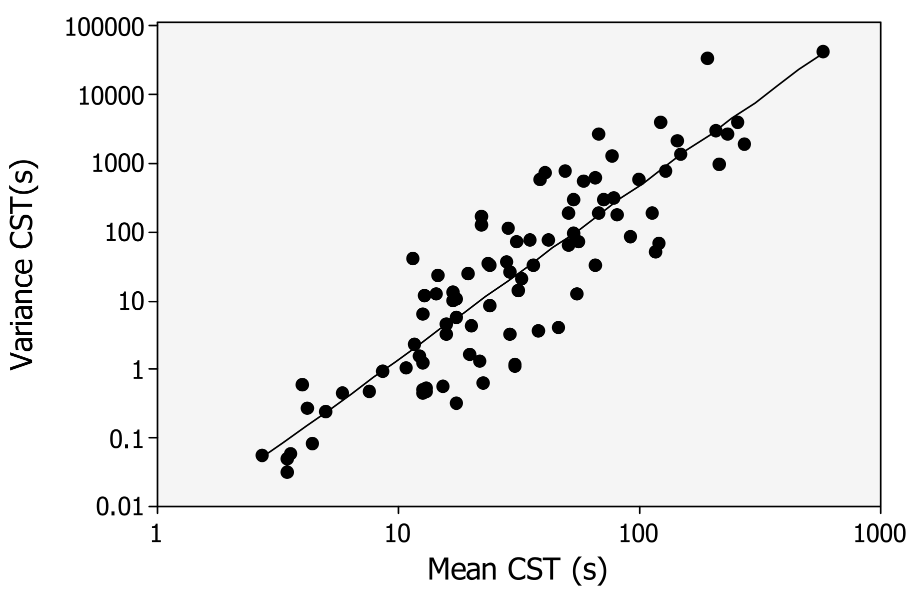
4.1.6). The longer the CST test times, the more variable the results were. Consequently, the variances increased linearly with respect to the mean CST estimates (Figure 4.1.1). This heteroskedacity could not be eliminated. The violations of the assumptions and the interactions meant that the results of the ANOVA were difficult to interpret.

Table 4.1.6 Tests for equality of variance among all combinations of factors and residual normality

Sludge	Levene's test statistic	p	Anderson-Darling test statistic	P
Surplus activated	8.82	0.000*	5.62	0.000*
Synthetic	2.38	0.003*	1.45	0.000*
Primary	7.88	0.000*	1.74	0.000*
Gully pot	13.93	0.000*	4.56	0.000*

* Significant at $\alpha = 0.01$

Figure 4.1.1 Relationship between the means and variances of CST estimates



To simplify the interpretation of the results, bar charts were constructed to provide a simple visual comparison of the trends in the means and coefficients of variations of the CST estimates when ranked in order of magnitude with respect to the types of sludge, the types of filter paper, and the three funnel geometries (Figures 4.1.2 to 4.1.9).

When testing gully pot sludge with 7 types of filter papers, the mean CST estimates tended to be consistently higher and more variable when using a circular 10 mm funnel than when using a circular 18 mm funnel and least using a rectangular funnel (Figures 4.1.2 and 4.1.3).

When testing primary sludge with 12 types of filter paper, the mean CST estimates tended to be consistently higher and more variable using a circular 18 mm funnel than when using a rectangular funnel (Figures 4.1.4 and 4.1.5).

When testing surplus activated sludge with 12 types of filter paper the mean CST estimates tended to be consistently higher and more variable when using a circular 10 mm funnel than when using a rectangular funnel (Figures 4.1.6 and 4.1.7). When testing synthetic sludge with 7 types of filter paper the mean CST estimates tended to be consistently higher and more variable using a circular 10 mm funnel than when using a rectangular funnel (Figures 4.1.8 and 4.1.9).

Figure 4.1.2 Mean CST estimates using gully pot sludge

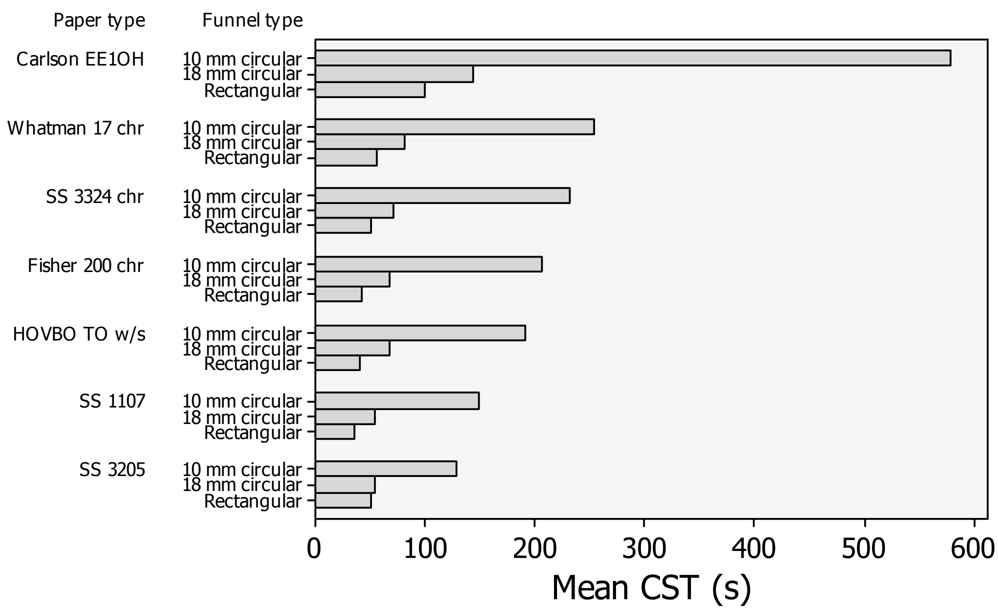


Figure 4.1.3 Coefficients of variation of CST using gully pot sludge

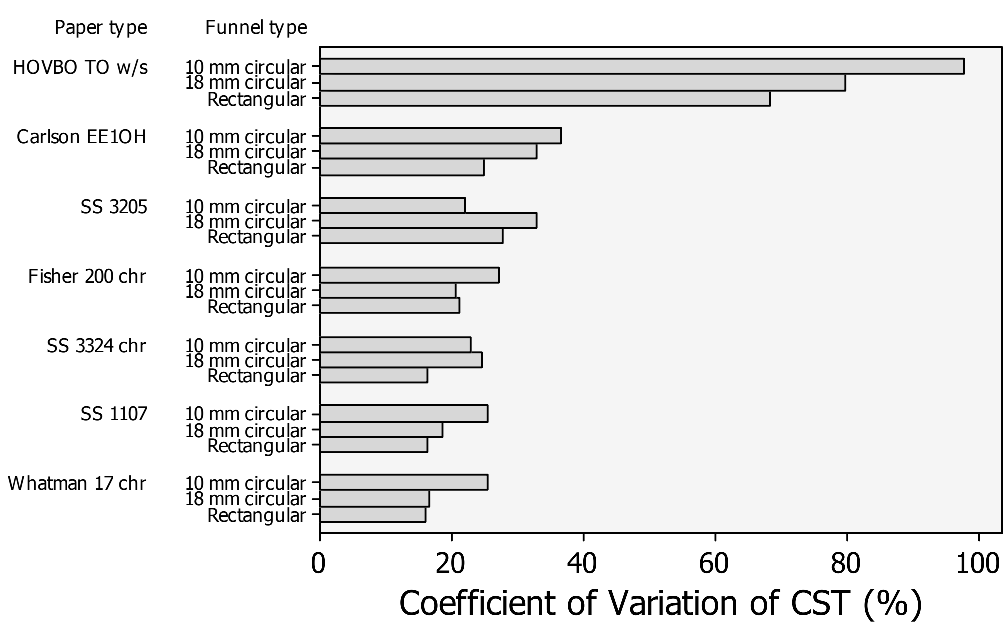


Figure 4.1.4 Mean CST estimates using primary sludge

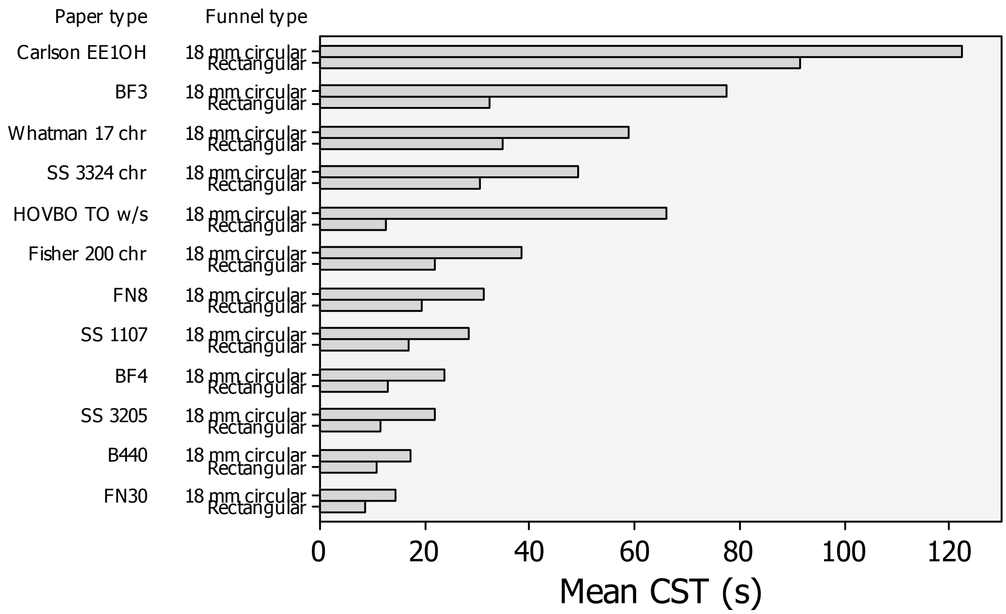


Figure 4.1.5 Coefficients of variation of CST using primary sludge

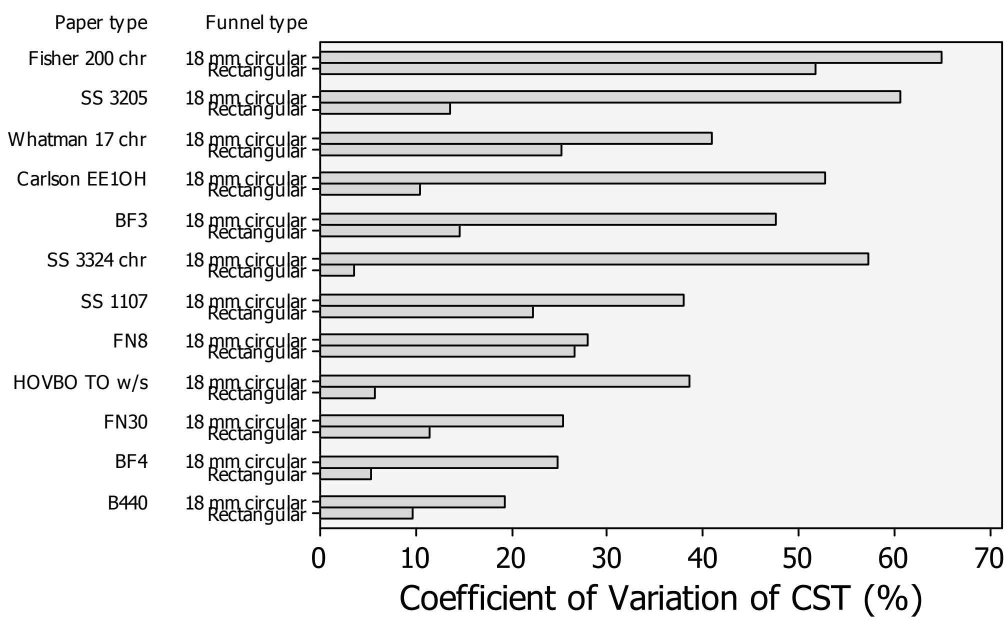


Figure 4.1.6 Mean CST estimates using synthetic sludge

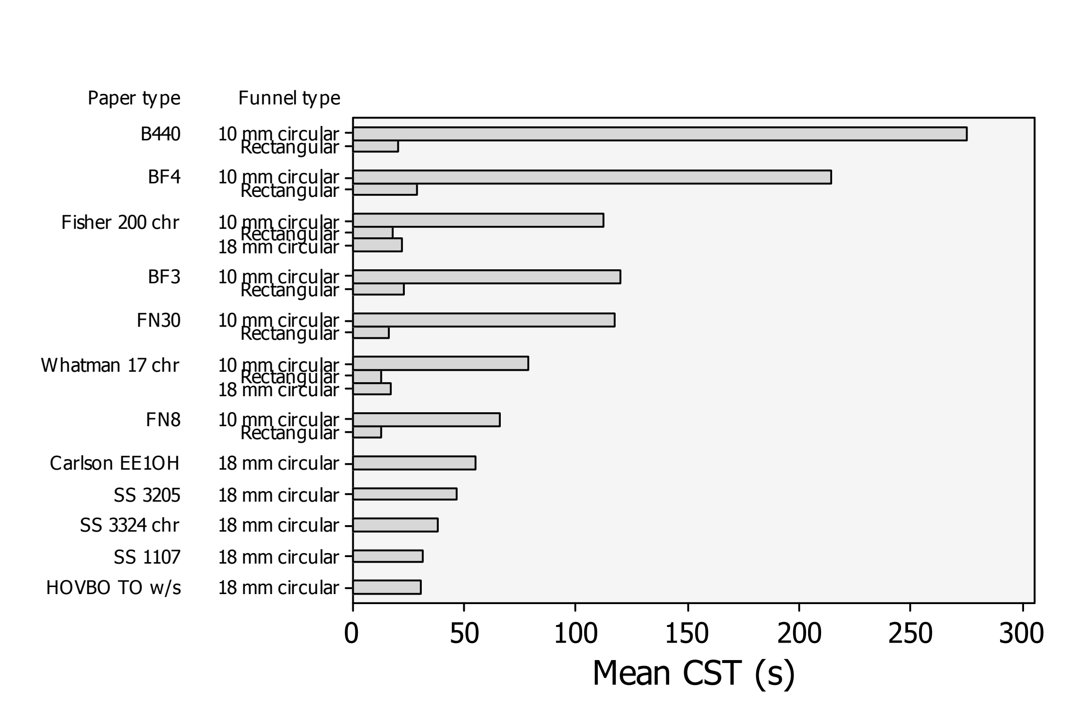


Figure 4.1.7 Coefficients of variation of CST using synthetic sludge

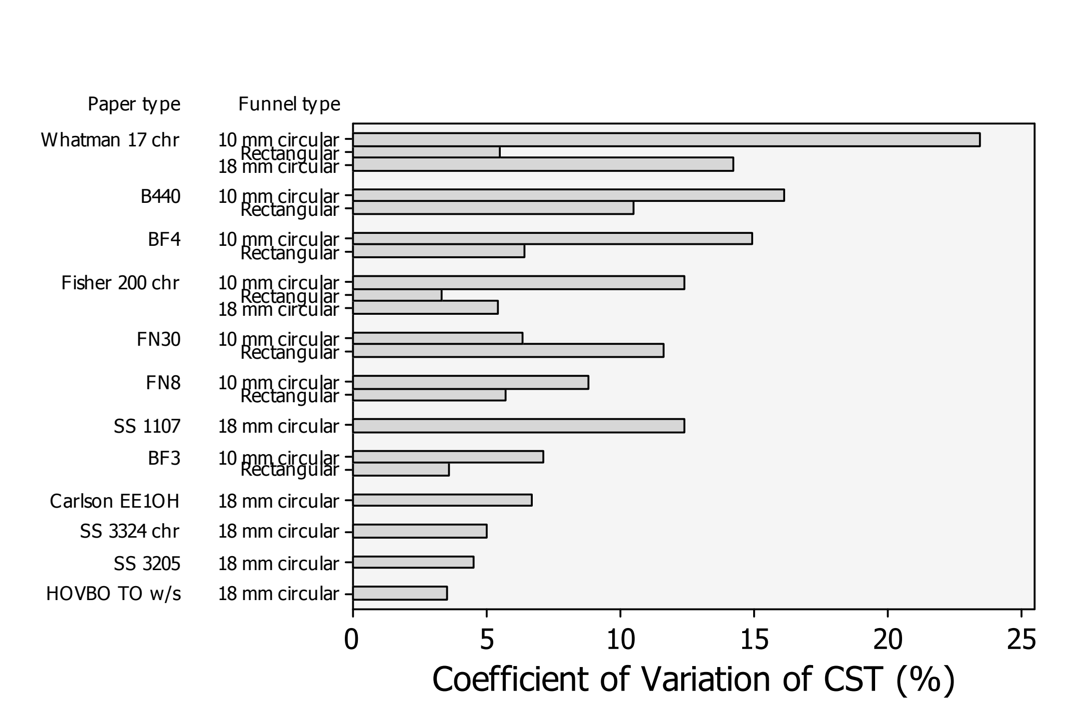


Figure 4.1.8 Mean CST estimates using surplus activated sludge

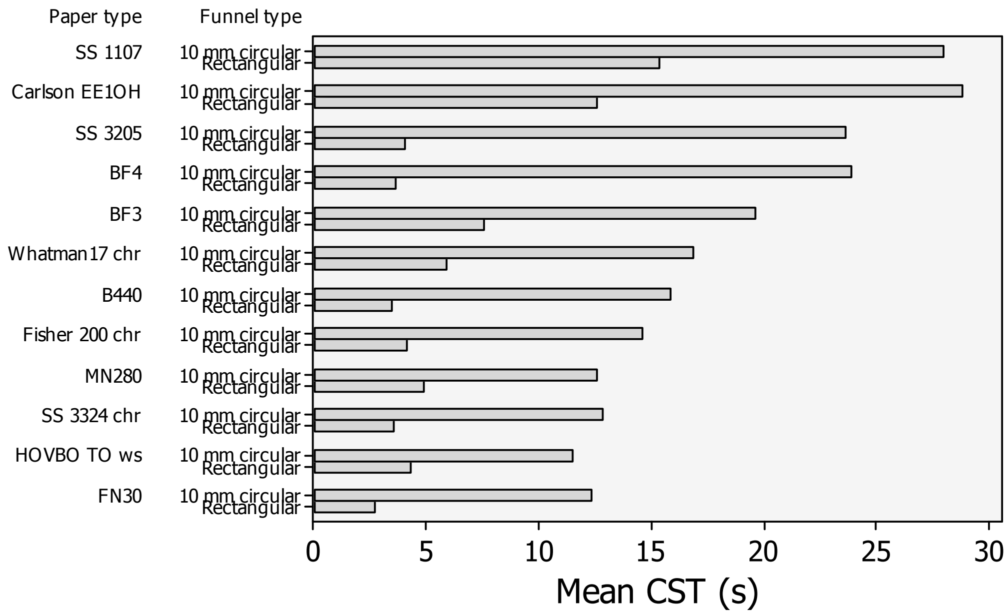
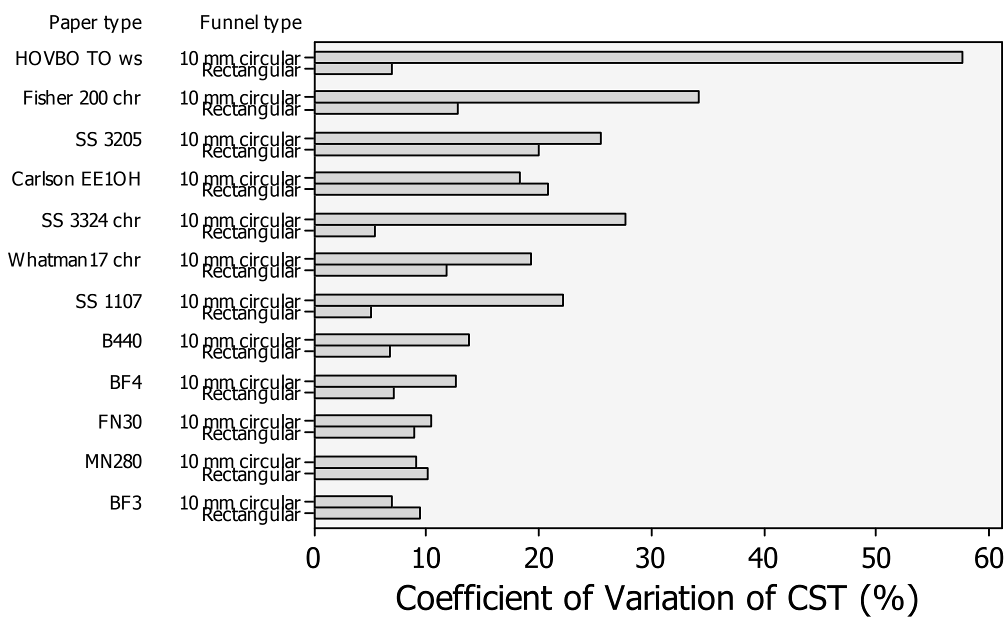


Figure 4.1.9 Coefficients of variation of CST using surplus activated sludge



One-way Kruskal-Wallis tests were done to identify differences between the median CST values by funnel geometry (Table 4.1.7) using the data in Table 4.1.4.

Table 4.1.7 Kruskal-Wallis tests to compare the median CST estimates with respect to funnel geometry and filter paper type

Sludge	Filter paper	Kruskal-Wallis χ^2 statistic	P
Gully pot	Carlson EE10H	48.0	0.000*
	Fisher 200 chr	57.1	0.000*
	HOVBO TO w/s	26.3	0.000*
	SS1107	57.1	0.000*
	SS3205	41.3	0.000*
	SS3324 chr	51.5	0.000*
	Whatman 17 chr	57.1	0.000*
Primary	B440	8.3	0.001*
	BF3	8.4	0.001*
	BF4	8.4	0.004*
	Carlson EE10H	0.1	0.754
	Fisher 200 chr	3.5	0.051*
	FN30	8.3	0.004*
	FN8	8.5	0.004*
	HOVBO TO w/s	5.0	0.025*
	SS1107	3.9	0.047*
	SS3205	2.5	0.117
	SS3324chr	0.5	0.465
	Whatman 17 chr	36.8	0.000*
Surplus activated	B440	29.4	0.000*
	BF3	29.3	0.000*
	BF4	29.4	0.000*
	Carlson EE10H	6.8	0.009*
	Fisher 200 chr	36.8	0.000*
	FN30	29.3	0.000*
	HOVBO TO w/s	6.8	0.009*
	MN280	29.3	0.000*
	SS1107	6.8	0.009*
	SS3205	6.9	0.009*
	SS3324 chr	6.9	0.009*
	Whatman 17 chr	36.8	0.000*
Synthetic	B440	6.8	0.009*
	BF3	6.8	0.009*
	BF4	6.8	0.009*
	Fisher 200 chr	12.5	0.002*
	FN30	6.8	0.009*
	FN8	6.8	0.009*
	Whatman 17 chr	12.5	0.002*

* Significant at $\alpha = 0.05$

The main assumptions of the Kruskal-Wallis test (that the minimum number of replicates within each factor combination should not be less than 5 and the CST test results can be logically ranked into an order of magnitude) were not violated. The null hypothesis was that the median CST estimates did not differ with respect to the three types of funnel geometry. This null hypothesis was rejected at $\alpha = 0.05$ in 35 out of the 38 Kruskal-Wallis tests (Table 4.1.7). The median CST estimates varied significantly at $\alpha = 0.05$ with respect to funnel geometry except when primary sludge was tested using Carlson EE10H paper ($p = 0.754$), SS3205 paper ($p = 0.117$), and SS3324 paper ($p = 0.465$).

Further experiments were carried out to explore the effect of the funnel geometry on the filtrate movement through the filter paper and to try to explain the observed differences in the CST values between circular and the rectangular funnels. The experiments were carried out by placing the funnel (either circular or rectangular) on a Whatman 17 chr filter paper (with and without using a sealant) using distilled water. The time required for the water front to move through a specified distance through the filter paper was recorded. Regular size filter papers were used with the circular funnel (70x90 mm) while a strip of filter paper (24x90 mm) was used for rectangular funnel. The funnel was placed at the centre of the filter paper which was marked at five equal distances from the centre of the circular funnel and from one of the edges of the rectangular funnel. The experiments were repeated five times for both the circular and the rectangular funnels with and without sealant (total number of runs, n was 20).

The experimental data were fitted based on the general model as in equation 2.4.1 (Hall and Hoff, 2002). This model describes the movement of the water front through porous media taking into consideration the effect of the funnel geometry. Equation 2.4.2 and equation 2.4.3 (which are special cases of the general model in equation 2.4.1) were used to plot the experimental data for the circular and the rectangular funnels, respectively.

For the circular funnel, figures 4.1.10 and 4.1.11 show the distance of the water front movement from the centre of the funnel to the power two minus the inner radius of the circular funnel (9mm) to the power two (D^2-r_o^2 (mm^2)) versus the square root of the time ($\sqrt{t}$) without and with using the sealant, respectively. The experimental data were fitted to the model in equation 2.4.2. The results showed that the water front movement through Whatman 17 chr filter paper using the circular funnel could be described by equation 4.1.1 without using the sealant and equation 4.1.2 with using the sealant.

$$y = 75 + 120.75x + 16.37x^2 \qquad (4.1.1)$$

$$y = 16.91 + 115.27x + 15.35x^2 \qquad (4.1.2)$$

where x= $\sqrt{t}$. These equations show a non linear relationship between the water front movement and the square root of the time (R^2 = 0.98 and 0.99 for without using the sealant and with using the sealant, respectively). This may indicate that the movement of the water front using the circular funnel (with and without using the sealant) advances non-linearly with the square root of the time.

Sorptivity divided by open porosity can be calculated from the results above. The sorptivity divided by the open porosity (S/f_e), which is the coefficient of the square root of the time in the model equation 2.4.2 divided by two×inner radius of the circular funnel (9mm), was 6.7 $mm.s^{-0.5}$ using the circular funnel without the sealant and 6.4 mm.s$^{-0.5}$ when the sealant was used. The sorptivity divided by the open porosity with using the sealant was lower than that without using the sealant. This may be used to support the hypothesis that using sealant reduces the leakage between the funnel and the filter paper (see section 4.4 for further details about applying the sealant to the CST test). The intercepts in the models represent the high absorption of filtrate at the beginning of the test (Hall and Hoff, 2002).

Figure 4.1.10 Distance² from the centre of the funnel minus the inner radius of the circular funnel² (D^2-r_o^2) versus square root of the time $\sqrt{t}$ for circular funnel without sealant

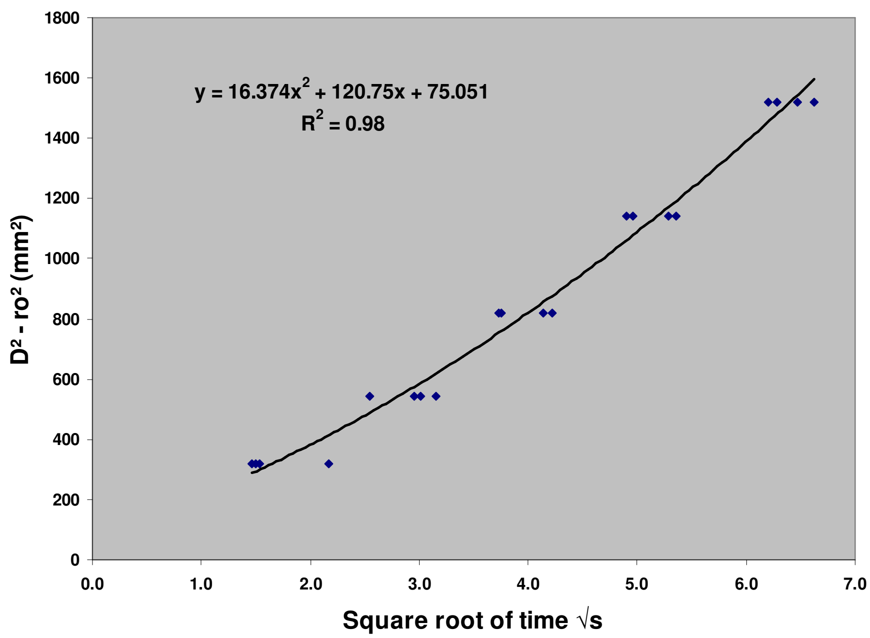
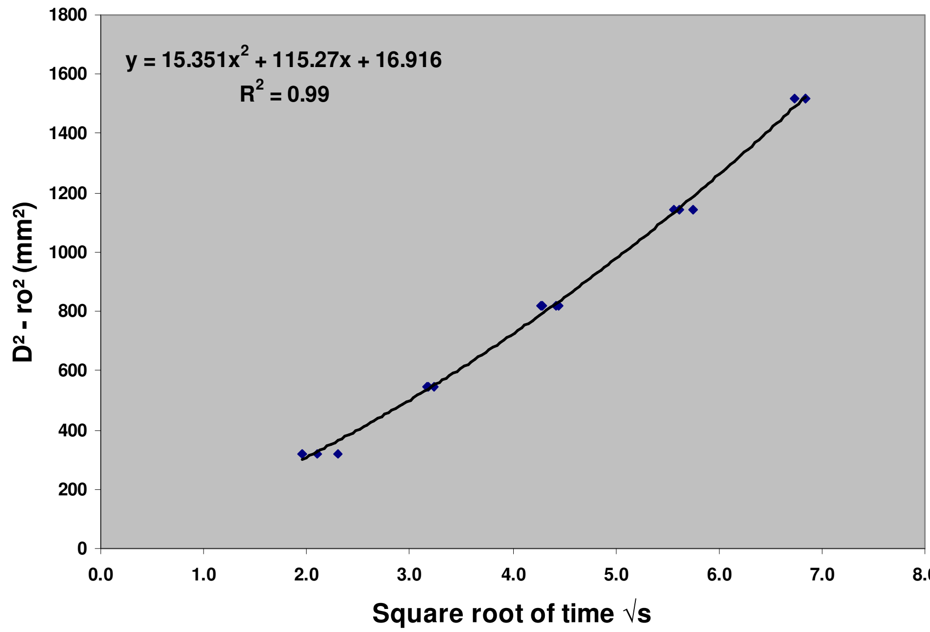


Figure 4.1.11 Distance² from the centre of the funnel minus the inner radius of the circular funnel² (D^2-r_o^2) versus square root of the time $\sqrt{t}$ for circular funnel with sealant



For the rectangular funnel, figures 4.1.12 and 4.1.13 show the distance (D in mm) versus the square root of the time ($\sqrt{t}$) for the rectangular funnel without and with using the sealant, respectively. The results showed that the water front movement through Whatman 17 chr paper could be describe by equation 4.1.3 without using the sealant and equation 4.1.4 with using the sealant.

$$y = 6.35x - 0.29 \qquad (4.1.3)$$

$$y = 5.84x - 0.92 \qquad (4.1.4)$$

where x=√t. The results showed almost a perfect fit (R^2 = 0.98 and 0.99 for without and with using sealant, respectively) for the experimental data to the linear model in Equation 2.4.3. This may indicate that the movement of the water front using the rectangular funnel (with and without using the sealant) advances linearly with the square root of the time. The results showed that the sorptivity divided by the open porosity (S/fe) value (coefficient of the square root of the time in the model equation 2.4.3) was 6.4 $mm.s^{-0.5}$ using the rectangular funnel without the sealant and it was 5.8 $mm.s^{-0.5}$ when the sealant was used. The sorptivity divided by the open porosity with using the sealant was lower than that without using the sealant. This shows again that the sealant may be effective in reducing the leakage between the funnel and the filter paper (see section 4.4 for further details about applying the sealant to the CST test).

Figure 4.1.12 Distance (D^2) from the edge of the rectangular funnel versus square root of the time $\sqrt{t}$ for rectangular funnel without sealant

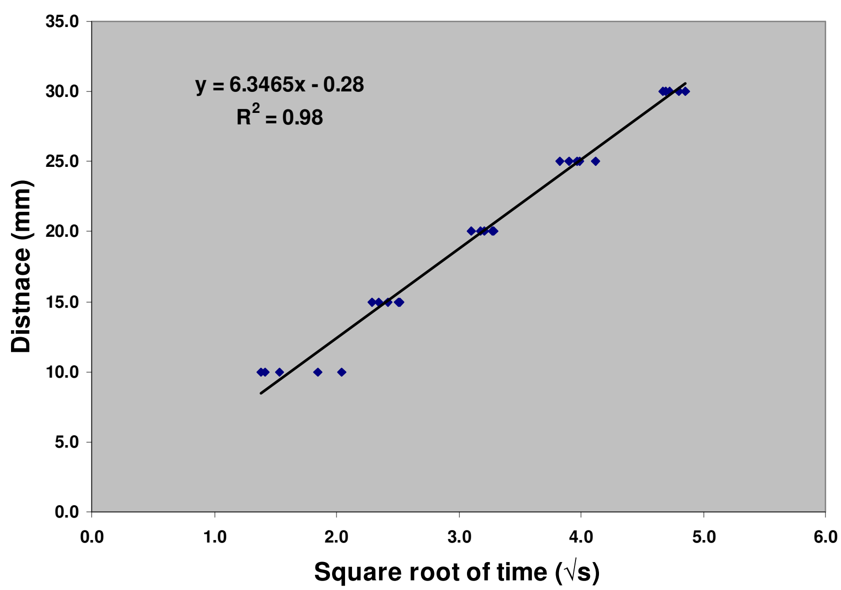
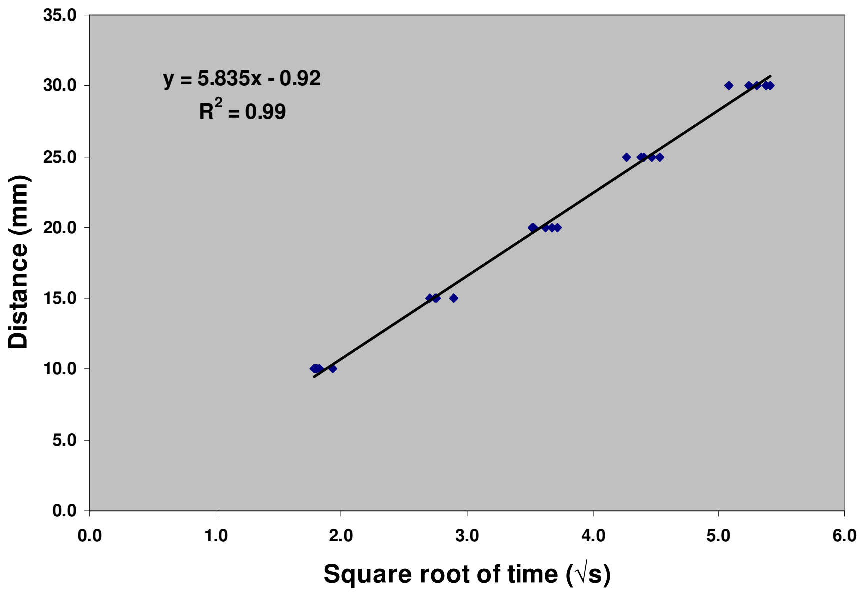


Figure 4.1.13 Distance (D^2) from the edge of the rectangular versus square root of the time $\sqrt{t}$ for rectangular funnel with sealant



4.1.4 Discussion

Due to the unbalanced design, deviations from residual normality, non-homogeneity of variance, and interaction effects, it was difficult to obtain an accurate interpretation using multi-factorial ANOVA and post-hoc multiple comparison tests. Nevertheless, there was clear visual evidence to conclude, by comparison of the means and coefficients of variation using bar charts, that the CST estimates were consistently the highest and most variable using the 10 mm circular funnels, smaller and less variable when using the 18 mm circular funnels, and consistently the lowest and least variable when using the rectangular funnels. The use of non-parametric Kruskal-Wallis tests to compare the median CST estimates also indicated a consistent trend. 35 out of the 38 Kruskal-Wallis tests indicated that the funnel geometry had a significant effect on the median CST estimates at $\alpha = 0.05$.

Irrespective of the type of filter paper used, the CST test results were consistently lower using rectangular funnels compared to circular funnels (except for three tests, when surplus sludge was tested using Carlson EE10H, MN280, and BF3 papers) and FN30 when synthetic sludge was used. It is concluded that the use of a rectangular funnel instead of the standard circular funnels when used with most types of filter paper significantly influenced the CST test results. When using 10 mm circular funnels the median (16 s to 188 s) and mean (17 s to 249 s) CST estimates were the highest. When using 18 mm circular funnels the median (32 s to 66 s) and mean (34 s to 77s) CST estimates were less. When using rectangular funnels the median (4 to 48 s) and mean (5 s to 53 s) were the lowest (Table 4.1.8).

Table 4.1.8 Descriptive statistics for CST tests (excluding the effects of filter papers)

Funnel	Sludge	Sample size	Minimum CST (s)	Maximum CST (s)	Median CST (s)	Mean CST (s)
10 mm circular	Surplus activated	175	6.3	36.4	15.8	17.1
	Synthetic	35	47	332	116.5	140.4
	Gully pot	140	60.6	1009.3	187.7	248.8
18 mm circular	Synthetic	35	14	56.6	31.7	34.2
	Primary	77	10.8	234.3	35.1	45.4
	Gully pot	210	20.9	227.2	66.1	76.7
Rectangular	Surplus activated	175	2.3	16.2	4.2	5.1
	Synthetic	35	11.4	31.4	17.2	18.6
	Primary	77	7.4	103.9	16	25.3
	Gully pot	140	11.3	149	48.7	53.6

The descriptive statistics excluding the effects of filter papers (Table 4.1.8) demonstrated that the advantage of using a rectangular funnel, irrespective of the type of filter paper, was especially apparent when testing heavy gully pot and primary sludges. The highest and most variable CST estimates were obtained when testing heavy gully pot sludge using small 10 mm diameter circular funnels (ranging from a minimum of 61s to a maximum of 1009 s). The CST estimates for gully pot sludge declined and were less variable when 18 mm diameter circular funnels were used (21 s to 227 s) but declined even further and were the least variable (11 s to 149 s) when rectangular funnels were used. These results confirmed the recommendation that the larger 18 mm diameter circular funnels, and preferably the rectangular funnels, should be used to test heavy sludges because the larger funnels significantly reduce the time taken to conduct the CST tests (Scholz, 2005).

One of the problems experienced during the standard CST test, particularly when testing heavy sludges with circular funnels, is that suspended solids and flocs accumulate on top of the filter paper by sedimentation. It follows that the heavy gully pot and primary sludges are expected to have longer CST testing times since they are influenced more by sedimentation in comparison to the lighter activated sludges. Longer testing times may also lead to an overestimation of the cake resistance, since the theory of the CST test does not take the effects of sedimentation into account (Scholz, 2005). Accordingly, the results of this study indicated that the median CST estimates using heavy sludges (gully pot and primary) were higher than those obtained using lighter sludges (surplus activated and synthetic). Since rectangular funnels reduce the sedimentation effect compared circular funnels (Tiller and Li, 2001) the CST estimates were consistently less when rectangular funnels were used relative to when circular funnels were used. The sedimentation is expected to be less when the rectangular funnel is used compared with the circular funnel due to the shorter testing time when the rectangular funnel is used.

Another reason for reduced CST times using rectangular funnels is that a

CST apparatus with a rectangular funnel should overcome the problem of anisotropic filter papers by making use of the flow in only one direction of the paper (Lee and Hsu, 1994b). When rectangular funnels are used, there should be a faster linear movement of the wet front through the filter papers, reducing the CST test times. This is also supported by the results of this study, as the movement for the experimental data using the rectangular funnel (with and without using the sealant) was best fitted using a linear relationship between distance travelled by the water front versus the square root of time. In contrast, when a circular funnel is used, the CST test time is higher, because the wet front has an elliptical shape, and its movement is slower and non-linear, particularly when anisotropic paper is used (Lee and Hsu, 1994b; Scholz, 2006). The results of this study agree with the results above and it was showed that the movement of the water front through the filter paper using the circular funnel can be described by a non-linear model.

4.2 The effects of the filter papers

4.2.1 Introduction

The aim was to explore the effects of different types of filter papers on the results of CST tests using natural and synthetic sludges in circular and rectangular funnels. The objectives were to compare the results of CST tests using isotropic and anisotropic papers, and to determine if alternative filter papers could be found that are cheaper and generate more consistent CST results than the standard Whatman 17 paper conventionally used for most CST tests. The sorptivity and the capillary suction pressure for Whatman 17 chr and Fisher 200 chr filter papers were measured and compared.

4.2.2 Experimental design

The same factorial design matrix defined in Table 4.1.3 was used. In view of the violations of the assumptions of multi-factorial ANOVA, multiple one-way Kruskal-Wallis tests were performed to determine if there were significant differences at $\alpha = 0.05$ between the median CST estimates with respect to selected filter paper types for each of the four sludge types and each of the three types of funnel geometry. The null hypothesis was that there was no difference between the median CST estimates measured using different types of filter papers for each combination of sludge and funnel geometry.

To determine which filters papers produced the lowest and highest CST test results, and which results were the least and most variable, the means and coefficients of variation of the CST test estimates obtained for each factor combination were sorted into rank order. This ranking method enabled the CST test results to be correlated with the pore diameters, the basis weights and the thicknesses of the filter papers using Spearman's rank correlation coefficients.

4.2.3 Results

4.2.3.1 Effect of filter papers on CST test results

The median CST test estimates with respect to filter paper type (Table 4.1.4) were compared using Kruskal-Wallis tests. The median CST estimates varied significantly at $p < 0.01$ with respect to the different filter paper types when tested with four types of sludges and three types of funnels (Table 4.2.1).

Table 4.2.1 Kruskal-Wallis tests to compare the median CST estimates with respect to filter paper types

Sludge	Number of filter papers tested	Kruskal-Wallis statistics					
		Circular 10 mm funnel		Circular 18 mm funnel		Rectangular funnel	
		χ^2	p	χ^2	P	χ^2	P
Gully pot	7	79.2	0.000*	99.8	0.000*	72.8	0.000*
Primary	12	-	-	49.6	0.000*	61.3	0.000*
Surplus activated	13	117.8	0.000*	-	-	158.8	0.000*
Synthetic	7	30.5	0.000*	32.5	0.000	31.2	0.000*

* Significant at $\alpha = 0.05$; - No data

Seven types of filter paper were ranked in order of magnitude with respect to the mean CST test results (Table 4.1.4). The mean CST estimates varied between anisotropic and isotropic filter papers, and whether the sludge was heavy or light. The isotropic Carlson EH10H papers were consistently ranked the lowest (6th or 7th) since these papers produced relatively high CST estimates using all types of sludge. The anisotropic Whatman 17 chr, SS3324 chr, and Fisher 200 chr papers were ranked 3rd to 7th using gully pot and primary sludges, reflecting relatively moderate to high CST estimates when testing heavy sludges. The isotropic HOVBO TO w/s, SS3205, and SS1107 papers were consistently ranked 1st to 4th when testing gully pot and primary sludges, reflecting that these isotropic papers tended to produce relatively lower CST estimates than the other papers when heavier sludges were tested. When testing lighter surplus activated sludge, however, the filter papers performed differently. The isotropic H0VB0 T0 w/s was ranked 1st, whilst the anisotropic Fisher 200 chr, SS324 chr, and Whatman 17 chr were ranked 2nd to 4th and the isotropic SS3205 and SS1107 were ranked 5th and 6th.

The coefficients of variation for the CST estimates were consistently low for the isotropic SS1107 filter paper, which was ranked 1st or 2nd when testing three types of natural sludge. In comparison, the anisotropic SS3324 was consistently ranked 3rd in the rank order. The variability in the CST estimates for the other filter papers did not appear to follow a consistent pattern.

Table 4.2.2 Filter papers ranked in order of means and coefficients of variation of CST estimates when testing three types of natural sludge

	Rank order of mean CST estimates			Rank order of coefficients of variation		
Filter paper	Gully pot	Primary	Surplus activated	Gully pot	Primary	Surplus activated
SS 3205 [i]	1	1	5	5	6	5
SS1107 [i]	2	2	6	2	2	1
HOVBO TO w/s [i]	3	4	1	7	1	7
SS 3324 chr[a]	5	5	2	3	3	3
Whatman 17 chr[a]	6	7	4	1	5	2
Fisher 200 chr[a]	4	3	3	4	7	6
Carlson EE10H [i]	7	6	7	6	4	4

Ranks: 1 = lowest to 7 = highest, [i] isotropic; [a] anisotropic; - no data

Table 4.2.3 Correlations between the physical properties of the filter papers and the rank orders of the means and coefficients of variation of the CST estimates

Sludge	CST Rank	Filter Thickness	Filter Weight	Filter Pore Diameter
Gully pot	Mean	0.943 *	0.865 *	-0.667*
	CV	0.086	0.108	-0.462
Primary	Mean	0.886*	0.703 *	-0.359
	CV	0.086	0.252	0.219
Surplus activated	Mean	0.143	0.090	-0.462
	CV	0.029	0.144	0.107

*Significant at $\alpha = 0.05$

Spearman's rank correlation analysis was used to test for linear relationships between the ranked physical properties of the filters (using the filter property data in Table 2.3.1) and the means and coefficients of variation of the CST estimates (using the ranks in Table 4.2.2). No significant rank correlations were found between the properties of the filter papers and the mean CST estimates when surplus activated sludge was tested, however significant correlations were found when testing heavier sludges (Table 4.2.3). The ranked CST estimates were negatively correlated with the ranks of the filter pore diameters using gully pot sludge (Spearman's rho = - 0.667). This negative correlation reflected that the highest CST test times were obtained using filter papers with small pore diameters (Carlson EE10H) whilst the lowest CST test times were obtained using filter papers with wider pore diameters (SS3205 and SS1107).

The ranked CST estimates using gully pot and primary sludges were positively correlated with the filter paper thicknesses and weights (Spearman's rho = 0.703 to 0.943). The positive coefficients reflected that the highest CST test times were obtained using thick heavy filter paper (Carlson EE10H) whilst the lowest CST test times were obtained using the thin light papers (SS33205 and SS1107). The CST test results ranked by coefficients of variation were not significantly correlated with the physical properties of the filter papers.

4.2.3.2 Measuring the sorptivity and the capillary suction pressure of filter papers

Sorptivity and capillary suction pressure were assessed for both Whatman 17 chr and Fisher 200 chr filter papers. Fisher 200 chr filter paper had been chosen to be compared with the standard filter paper as it has similar physical properties such thickness, basis weight, chromatographic type, and flow rate. The mean CST values for Fisher 200 chr filter paper had been shown to mostly have lower values when compared with those of Whatman 17 chr filter paper (Table 4.2.2).

The methods described by Meeten and Lebreton (1992) and Meeten and Smeulders (1995) were used to compare the sorptivity and capillary suction pressure of Whatman 17 chr and Fisher 200 chr filter papers. A distance of two cm of the end of a rectangular strip of filter paper (30 cm long x 2.5 cm wide) was dipped into a flask of water. The strip was marked at 1 cm intervals. The time required for the wet water front to rise above the water reservoir was recorded at 1 cm intervals. To reduce interference by evaporation, the filter paper was fitted inside the centre of a transparent plastic tube (making sure the filter paper did not touch the sides) while carrying out the experiment. The measurements were replicated five times for each filter paper type.

The rising of the wet water front (i.e. the rate of increase in the height of water on the filter paper) was defined by equation 4.2.1.

$$\frac{dy}{dt} = \frac{B}{A^2 y} - \frac{B}{A} \qquad (4.2.1)$$

Where A = ρ g/P; B = k ρ^2 g^2/ μ ε P; ρ and g are the liquid density (kg/m^3) and the gravitational force per unit weight respectively (kg/m^2). The sorpitivity (sorptivity /open porosity) was calculated as the slope of the line of distance of the rising water front (mm) versus the square root of time ($\sqrt{s}$) as I = s $\sqrt{t}$, where I is the cumulative filtrate volume.

The capillary suction pressure (Pa) was calculated from the slope and the intercept of the line. The time of the rising of the wet water front was analysed by plotting dy/dt versus 1/y and extrapolated to obtain the maximum height y_{max} (y_o)

following the same procedure and using the same model (equation 4.2.1) as that of Meeten and Smeulders (1995). The gradient function dy/dt was calculated using Matlab 6 for both Whatman 17 chr and Fisher 200 chr filter papers.

The following are detailed explanations of the procedure that was followed in this study to measure the capillary suction pressure.

Equation 4.2.1 was simplified to equation 4.2.2.

$$\frac{dy}{dt} = \frac{m}{y} + c \qquad (4.2.2)$$

where

$$m = \frac{B}{A^2} \text{ and } c = -\frac{B}{A}$$

$$\frac{m}{c} = -\frac{B}{A^2}\frac{A}{B} = -\frac{1}{A} \qquad (4.2.3)$$

$$\frac{dy}{dt} = 0.0 \text{ then}$$

$$\frac{m}{y_o} = -c \qquad (4.2.4)$$

$$y_o = -\frac{m}{c} \qquad (4.2.5)$$

$$\frac{1}{y_o} = -\frac{c}{m} = A \qquad (4.2.6)$$

The sorptivity values are presented in Figures 4.2.1 and 4.2.2 for Whatman 17 chr and Fisher 200 chr, respectively. The mean and coefficient of variation of sorptivity were 3.2 $mm.s^{-0.5}$ and 2.4% for Whatman 17 chr and 4.3 $mm.s^{-0.5}$ and 6.0% for Fisher 200 chr filter papers, respectively. Sorptivity and CST are inversely related where the CST is expected to be lower for papers with higher sorptivity values. The mean sorptivity for Fisher 200 chr was higher than that for Whatman 17 chr and so it is expected that the CST values for Fisher 200 chr should be lower than those for Whatman 17 chr and this was supported by the CST results in Table 4.2.2. The coefficient of variation of CST was in agreement with that of sorptivity (i.e. lower CV of both CST and sorptivity for Whatman 17 chr compared with Fisher 200 chr filter paper).

Figure 4.2.1 Plot of distance versus square root of time measuring sorptivity of Whatman 17 chr filter paper

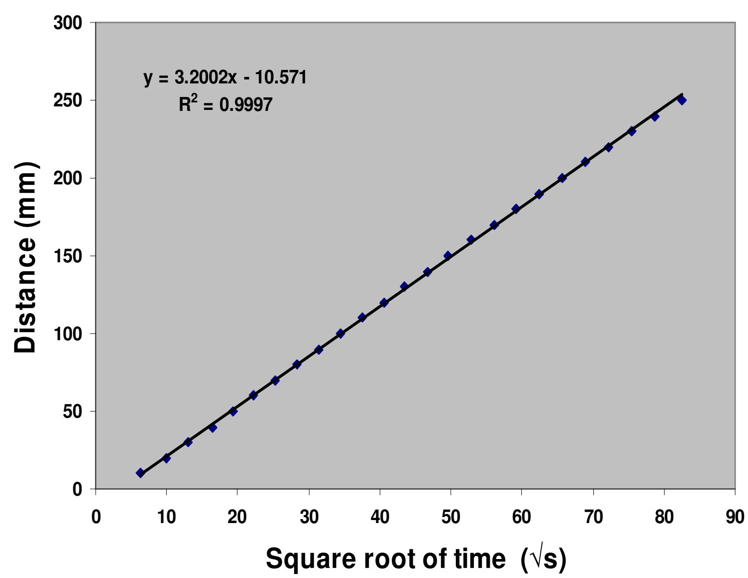
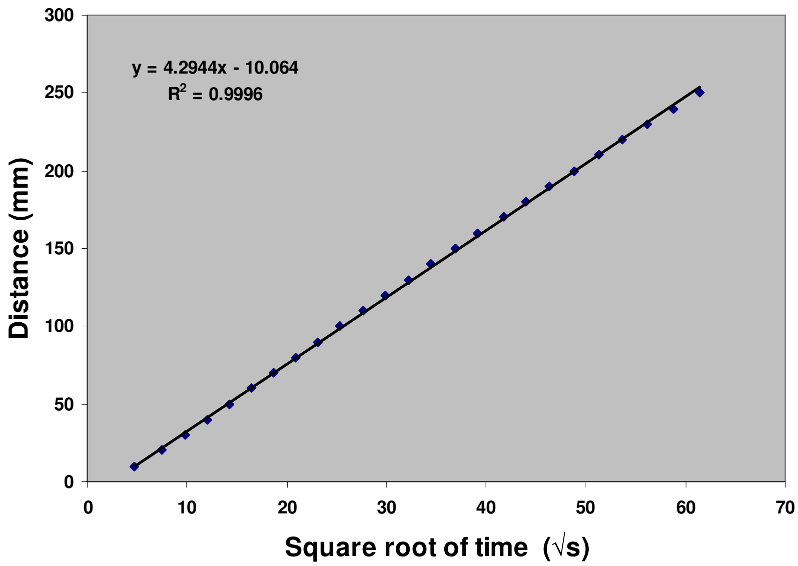


Figure 4.2.2 Plot of distance versus square root of time measuring sorptivity of Fisher 200 chr filter paper



The capillary succession pressure for Whatman 17 chr filter paper was calculated based on the values of $\frac{1}{y_o} = 0.001$ 1/mm and y_o= 1000 mm (Figure 4.2.3). The capillary suction pressure is defined as p = ρ g/A or ρ g y_o (as shown before). By using ρ = 1000 Kg/m^3 and g = 9.8 m/s^2 the capillary suction pressure is 9800 Pa.

The same procedure was followed for Fisher 200 chr filter paper using $\frac{1}{y_o} = 0.002$ 1/mm and y_o = 500 mm (Figure 4.2.4). The capillary suction pressure for Fisher 200 chr filter paper (p = ρ g y_o) = 4900 Pa.

Figure 4.2.3 Plot of gradients dy/dt (mm/ s) versus 1/y (1/mm) of capillary rise of water front in Whatman 17 chr filter paper strip

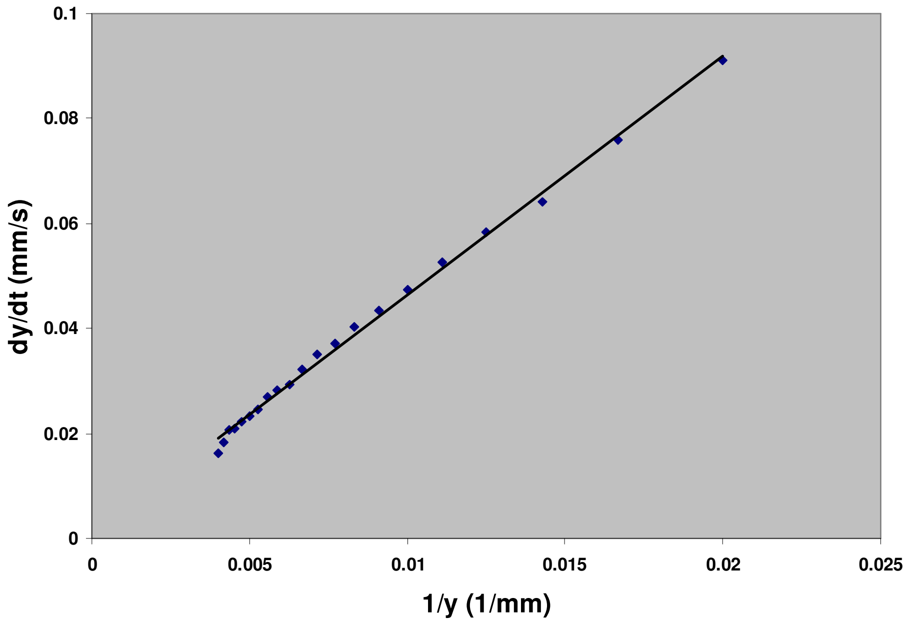
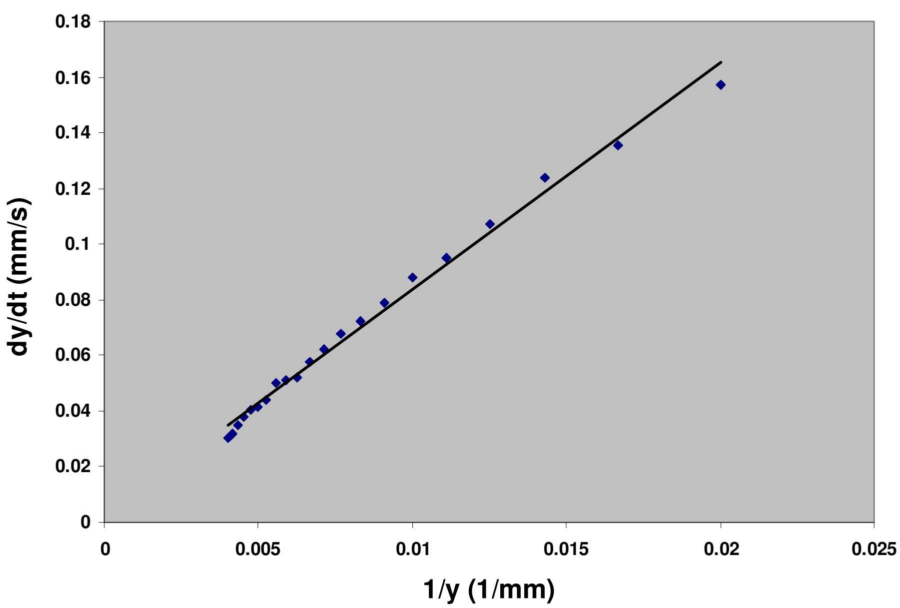


Figure 4.2.4 Plot of gradients dy/dt (mm/ s) versus 1/y (1/mm) of capillary rise of water front in Fisher 200 chr filter paper strip



4.2.5 Discussion

The effects of different filter paper types on the results of CST tests were compared, bearing in mind that the overall controlling influence was funnel size, (section 4.1). The trends in the results indicated that the filter paper type significantly influenced the results of the CST tests when funnel size was taken into account. The effects of the filter papers were particularly important when testing heavy sludges.

The anisotropic property of some filter papers, due to the presence of a longitudinal grain, causes the filtrate to move faster in one direction along the grain than across, whilst in isotropic papers, this different is not apparent. It has been suggested that the uneven movement of fluid across the diameter of anisotropic filters influences the results of CST tests (Lee and Hsu, 1992). The current study demonstrated, however, that there was no clear and absolute difference between the CST test results using isotropic and anisotropic papers. The isotropic papers HOVBO3 TO w/s, SS3205, and SS1107 generally produced lower CST estimates ranked 1st to 4th when testing gully pot and primary sludges, confirming that low CST test times are associated with some isotropic papers. Carlson EE10H, however, is also an isotropic paper, but it was associated with high CST estimates, ranked 6th to 7th in the order of magnitude. The CST test results using Carlson EE10H were consistently higher than the results using anisotropic papers, including both Fisher 200 chr and Whatman 17 chr. It is considered therefore, that the effects of filter papers on the results of CST tests are not only divided by their isotropic or anisotropic properties. Other physical properties of filters may influence CST test results including their porosity, weight, and thickness.

When testing heavy sludges, the ranked CST estimates were negatively correlated with the ranked pore sizes of the filters, and positively correlated with the ranked weights and thicknesses of the filters. The highest CST estimates were obtained using a thick heavy filter with small pore diameter (i.e. Carlson EE10H) when testing primary and gully pot sludges. When testing heavy sludges using thick heavy filter papers with small pores, irrespective of whether or not the paper is isotropic or anisotropic, the CST may be elevated due to the high resistance to filtration associated increase in resistance to flow of filtrate within smaller pores, sedimentation, and dense cake formation (Yukseler, *et al.,* 2007). The CST may be lower when testing heavy sludges using thin light filter papers with a larger pore size (e.g. SS33205 and SS1107) due to the lower resistance to filtration associated with the lower filtration resistance in larger pores, less sedimentation, and less cake formation. The correlation between filter paper pore size, thickness, and weight, and the results of CST tests was, however, not apparent when testing lighter surplus activated sludges, for which blockage, sedimentation, and cake formation are less of a problem than when testing heavy sludges. No correlation was found between the filter paper pore size, thickness, and weight, and the coefficients of variation of the CST test estimates. It might be inferred that the physical properties may not necessarily influence the variability in the results. However, this conclusion has to be taken with caution as the data used for this simple correlation analysis was small and due to the nature of the variables, the underlying assumptions might not be satisfied (most importantly normality and linearity).

Scholz (2005) recommended that research should be undertaken to determine if filter papers other than Whatman 17 chr (the standard filter paper used in most

CST tests) might be more useful, particularly for specific applications e.g. different types of sludge. The results of this study confirmed that Whatman 17 chr did not produce the most consistent results in the shortest time. The CST estimates were frequently higher and more variable between replicates when using Whatman 17 chr papers than when using other filter papers.

The paper exhibiting the most desirable combination of low price, consistently low CST test times and variability between replicates when testing heavy sludges, was SS1107, a thin light isotropic paper. The isotropic paper, H0VB0 T0 w/s was found to produce higher CST estimates when testing heavy gully pot sludge, but lower times with lighter sludges.

It might be concluded that Whatman 17 chr papers could be replaced by isotropic filter papers, in order to lower the cost, reduce the CST test times and improve the precision of the estimates. SS1107 is considered to be suitable for testing heavy sludges.

Sorptivity and capillary suction pressure were estimated and compared for Whatman 17 chr and Fisher 200 chr filter papers. The sorptivity of Whatman 17 chr paper was lower than that of Fisher 200 chr which is in agreement with the CST results found earlier (mean CST values for Whatman 17 chr were higher than those for Fisher 200 chr). The sorptivity measures the flow rate of filtrate inside the filter paper as sorptivity is function of the filter paper permeability and the capillary suction pressure. The flow rate through the filter paper depends on both the capillary suction pressure and the pore structure (Hall and Hoff, 2002). With smaller pore diameter, the capillary suction pressure increases but the flow rate becomes lower. This is due to increase in resistance to flow of filtrate within smaller pores (personal communication with Professor Chris Hall, Prof. of Materials, University of Edinburgh, June 2010).

The capillary suction pressure depends on the surface tension and the diameter of the pores. The capillary suction pressure = $2\sigma / r$ where σ is the surface tension and r is the pore radius (Hall and Hoff, 2002). Although the capillary suction pressure for Whatman 17 chr was higher than that for Fisher 200 chr, the CST values were lower for Fisher 200 chr than Whatman 17 chr which could be due to the differences in pore size and structure as discussed earlier. However, there is no available record about the pore size of Fisher 200 chr filter paper. The capillary suction pressure for Whatman 17 chr (9.80kpa) was within the range of the values reported in other studies: 10.00 kpa (Baskerville and Gale, 1968), 4.90 kpa (Tiller *et al.,* 1990), 7.47 kpa (Meeten and Lebreton, 1992), and 6.40 kpa (Meeten and Smeulders, 1995).

4.3 The effects of stirring

4.3.1 Introduction

The aim of this experiment was to investigate the effects of stirring activity (a current induced by a stirrer within the sludge chamber) on the results of the CST tests. The purpose of stirring was to alleviate or reduce the unfavourable effect of sedimentation, which is considered to be particularly problematic when testing heavy slow filtering gully pot and primary sludges.

4.3.2 Experimental design

The effects of stirring on the results of CST tests were explored using 3 sludge types and 7 filter papers types (Table 4.3.1). The funnel geometry was constant. A circular 18.0 mm funnel, with or without the stirrer, was used for each test. All tests were performed at 20 ±1^{o}C.

Table 4.3.1 Factorial design matrix to in investigate the effects of stirring

Stirring	Paper	Sludge		
		Gully pot	Primary	Synthetic
No	Carlson EE1OH	50	10	5
	Fisher 200 chr	50	20	10
	HOVBO TO w/s	50	10	5
	SS 1107	50	10	5
	SS 3205	50	10	5
	SS 3324 chr	50	10	5
	Whatman 17 chr	50	20	10
Yes	Carlson EE1OH	10	5	5
	Fisher 200 chr	20	10	5
	HOVBO TO w/s	20	5	5
	SS 1107	10	5	5
	SS 3205	10	5	5
	SS 3324 chr	10	5	5
	Whatman 17 chr	20	10	5

To optimize the power of statistical analysis there should ideally be an equal number of replicates for each factor combination; however, the number of replicates in each cell of the factorial design matrix (Table 4.3.1) varied from 5 to 50. Nevertheless, a three-way unbalanced ANOVA assuming a General Linear Model was justifiable to test for the main effects (sludge, paper, and stirring) and the interaction effects (sludge x paper, sludge x stirring, paper x stirring, and sludge x paper x stirring). The Anderson-Darling tests (Table 4.3.2) indicated that the residuals deviated strongly from normality at $p < 0.01$, and the p values < 0.01 for

Levene's test indicated that the variances were not equal across all combinations of factors. Therefore, logarithmic transformations were performed, following the results of Box-Cox tests, to help normalize the data.

4.3.3 Results

The descriptive statistics for the results of the CST tests to investigate the effects of stirring are presented in Table 4.3.3

The null hypotheses of ANOVA that the sludges, filter papers, and stirring had no effects on the logarithmically transformed mean CST estimates were rejected with respect to the sludges and papers, indicated by p values < 0.01 for the F statistics; however stirring had no significant effect on the mean CST estimates, indicated by $p = 0.133$ for the F statistic (Table 4.3.4). There was also a significant interactions between the filter paper types, the sludge types, and stirring, indicated p < 0.01 for the interaction terms (Sludge x Paper, Sludge x Stirring, Paper x Stirring, Sludge x Paper x Stirring).

Table 4.3.2 Tests for equality of variance and residual normality

Sludge	Levene's test statistic	p	Anderson-Darling test statistic	P
Gully pot	18.11	0.000	2.41	0.000*
Primary	6.39	0.000	1.21	0.000*
Synthetic	4.41	0.000	0.89	0.022*

*Significant at α =0.05

Table 4.3.3 Descriptive statistics for the CST tests (effects of stirring)

Paper	Stirring	Min	Max	Median	Mean	SD	CV %
Sludge: Gully pot							
Carlson EE1OH	No	67.4	227.2	115.6	125.9	44.9	35.7
	Yes	122.8	204.5	143.2	151.2	25.1	16.6
Fisher 200 chr	No	31.9	105.1	56.2	57.1	17.5	30.6
	Yes	35.2	78.6	56.9	55.8	13.2	23.6
HOVBO TO w/s	No	11.3	204.6	36.8	56.5	46.6	82.5
	Yes	17.8	163.8	39.1	67.2	50.9	75.8
SS 1107	No	26.6	80.2	44.3	46.4	12.1	26.0
	Yes	38.2	62.7	50.1	50.5	8.1	16.1
SS 3205	No	25.6	112.7	50.9	52.5	16.2	30.8
	Yes	31.6	49.2	38.6	39.5	5.3	13.5
SS 3324 chr	No	32.1	105.2	58.7	63.2	17.7	27.9
	Yes	68.3	84.4	78.2	77.6	5.8	7.5
Whatman 17 chr	No	37.3	105.7	68.3	70.7	17.2	24.3
	Yes	49.0	89.8	63.8	69.6	14.2	20.4
Sludge: Primary							
Carlson EE1OH	No	79.9	234.3	90.2	107.0	46.6	43.5
	Yes	100.5	184.9	129.7	141.8	38.7	27.3
Fisher 200 chr	No	10.4	72.1	19.1	30.2	20.7	68.5
	Yes	38.7	114.5	62.3	70.9	27.5	38.8
HOVBO TO w/s	No	11.7	93.2	22.8	39.3	32.9	83.7
	Yes	27.5	38.0	32.6	32.9	3.8	11.5
SS 1107	No	14.0	43.9	20.1	22.6	9.8	43.5
	Yes	26.5	68.0	37.0	41.9	16.2	38.7
SS 3205	No	9.8	44.5	12.35	16.8	10.5	62.8
	Yes	8.4	40.3	25.7	26.0	13.3	51.0
SS 3324 chr	No	22.4	90.2	30.85	39.8	21.2	53.2
	Yes	65.8	95.8	73.5	77.6	11.8	15.3
Whatman 17 chr	No	19.5	118.0	40.95	46.9	21.6	46.0
	Yes	57.3	307.4	161.4	169.0	106.8	63.2
Sludge: synthetic							
Carlson EE1OH	No	48.1	56.6	56.2	54.6	3.6	6.7
	Yes	17.0	19.5	18.6	18.3	1.0	5.5
Fisher 200 chr	No	16.9	23.6	19.4	19.6	2.4	12.3
	Yes	12.4	13.8	13.0	13.0	0.5	4.2
HOVBO TO w/s	No	29.3	32.1	30.2	30.5	1.1	3.5
	Yes	12.4	16.8	16.1	15.5	1.8	11.4
SS 1107	No	28.3	37.6	29.5	31.1	3.9	12.4
	Yes	12.5	17.9	14.8	14.9	2.1	13.8
SS 3205	No	42.6	47.9	46.6	46.0	2.1	4.5
	Yes	26.3	31.4	26.7	27.8	2.1	7.6
SS 3324 chr	No	36.0	40.5	39.0	38.2	1.9	5.0
	Yes	22.0	25.4	23.6	23.8	1.3	5.5
Whatman 17 chr	No	11.4	19.4	13.6	14.9	3.0	20.3
	Yes	19.0	21.1	20.4	20.3	0.8	3.9

Min, Minimum; Max, Maximum, SD, Standard Deviation, CV%, Coefficient of Variation

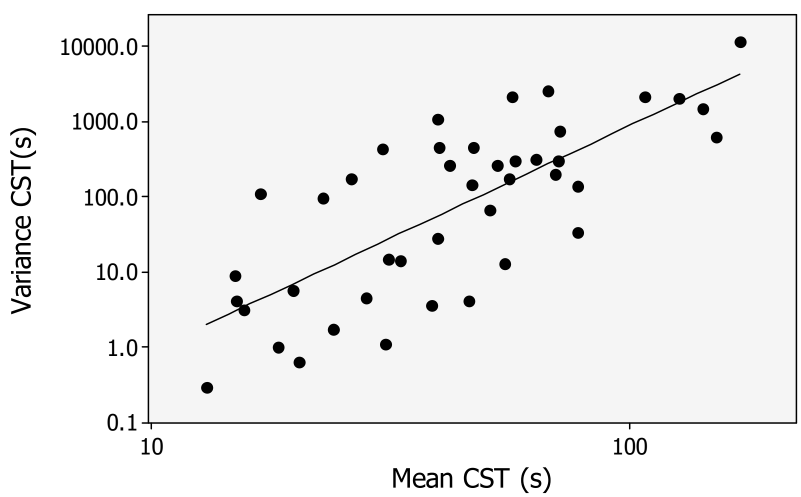
Table 4.3.4 Three-way ANOVA to compare the mean CST estimates with respect to sludge type, filter paper type, and stirring

Source of variance	Type III Sum of Squares	Degrees of Freedom	Mean Square	F statistic	P
Sludge	10.509	2	5.255	179.910	0.000*
Paper	6.478	6	1.080	36.963	0.000*
Stirring	0.066	1	0.066	2.263	0.133
Sludge x Paper	4.086	12	0.341	11.658	0.000*
Sludge x Stirring	2.788	2	1.394	47.721	0.000*
Paper x Stirring	0.922	6	0.154	5.262	0.000*
Sludge x Paper x Stirring	1.215	12	0.101	3.467	0.000*
Error	18.313	627	0.029		
Total	1971.201	669			

* Significant at $\alpha = 0.01$

The longer the CST test times, then the more variable were the results. The variances increased linearly with respect to the mean CST estimates (Figure 4.3.1).

Figure 4.3.1 Relationship between the means and variances of CST estimates



In order to simplify the interpretation of the data (without the need for data transformation), bar charts were constructed to provide visual comparisons of the trends in the mean CST estimates when ranked in order of magnitude with respect to the types of sludge, types of filter paper, and whether or not the samples were stirred or unstirred (Figures 4.3.2 to 4.3.4).

Figure 4.3.2 Effects of stirring on results of CST tests using gully pot sludge

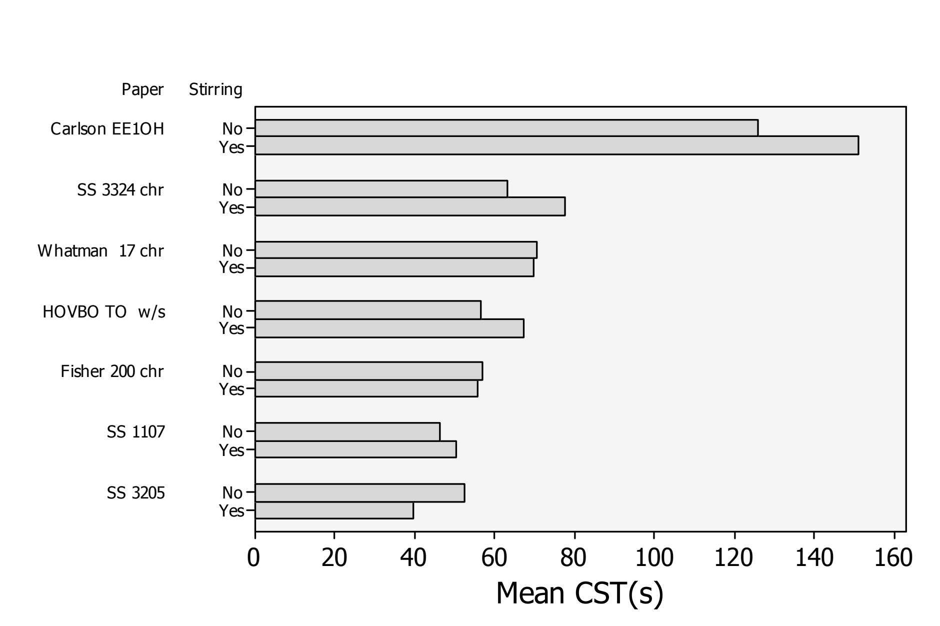


Figure 4.3.3 Effects of stirring on results of CST tests using primary sludge

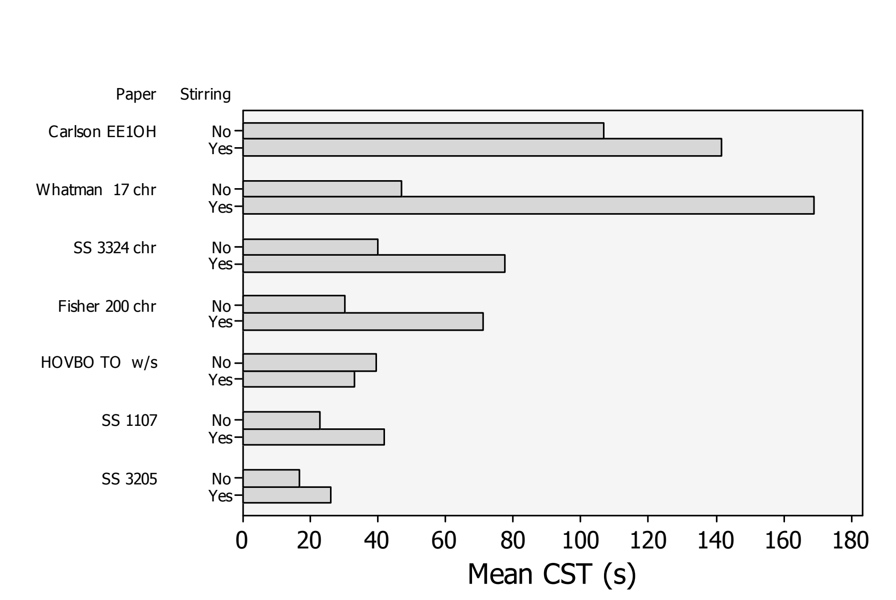
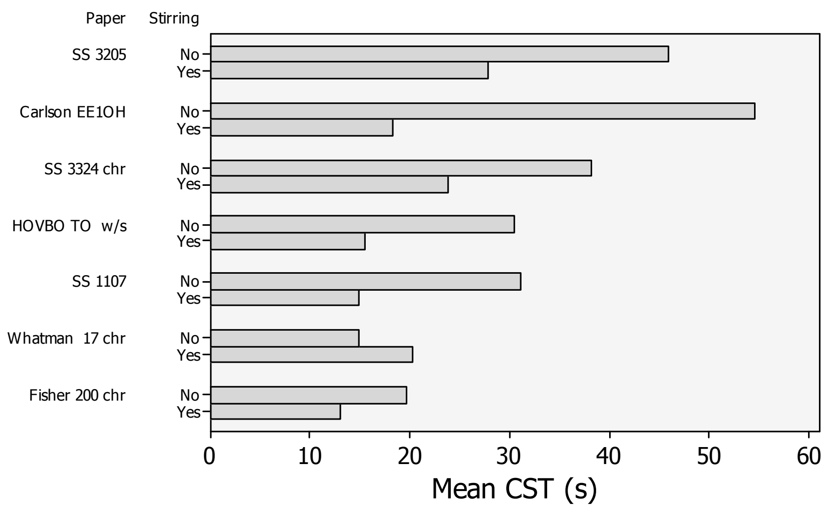


Figure 4.3.4 Effects of stirring on results of CST tests using synthetic sludge



When Carlson EE10H, SS33234 chr, and SS1107 w/s papers were used to test gully pot sludge, the mean CST estimates were higher when the samples were stirred than when the samples were not stirred. When the primary sludge were tested, the mean CST estimates obtained using Carlson EE10H, SS3205, SS33234 chr, and SS1107 w/s papers were consistently higher when the samples were stirred than when the samples were not stirred. When Carlson EE10H, SS3205, SS33234 chr, and SS1107 w/s papers were used to test synthetic sludge, however, the mean CST estimates were less when the samples were stirred compared with when the samples were not stirred. This pattern with respect to the effect of stirring was not, however observed using HOVBO TO w/s, Whatman 17 chr, and Fisher 200 chr papers.

4.3.4 Discussion

Scholz (2005) suggested that a current induced by a stirrer within the sludge chamber, may reduce or prevent sedimentation, thereby improving the results of CST tests. It was also suggested that stirring may have a greater influence on the results of the CST test when using Whatman 17 chr than when using other papers such as Fisher 200 chr. The results presented here are not consistent with these suggestions. Consequently the potential beneficial effects of stirring on the results of the CST tests could not be unequivocally proved.

It is possible that the current induced by the stirrer was insufficient to reduce or prevent sedimentation, particularly when testing the heavy gully pot and primary sludges, explaining why stirring had no statistically significant effects.

4.4 The effects of a sealant

4.4.1 Introduction

The aim of this experiment was to determine whether or not a sealant applied to the funnel influenced the results of CST tests. The experiments were carried out using a range of synthetic sludge concentrations, two types of filter paper, and two types of funnel.

4.4.2 Experimental design

The replicated experimental design explored the effects of three factors and one covariate upon the mean CST values. The covariate comprised five synthetic sludge concentrations at 2.30, 5.64, 8.80, 12.10, and 15.30 g/l. The three factors were

(1) with or without sealant; (2) two types of filter paper i.e., Whatman 17 chr or Fisher 200 chr; and (3) two types of funnel i.e., circular with 18 mm diameter or rectangular (equivalent in size to 18 mm diameter circular funnel). The samples were not stirred, and all the observations were made at 20±1°C.

4.4.3 Results

The means and coefficients of variation of the CST estimates for the eight combinations of factors at the five sludge concentrations are presented in Table 4.4.1.

Table 4.4.1 Comparison of means and coefficients of variation of CST (s) with respect to three fixed factors: (1) without or with sealant; (2) two types of filter paper (Fisher 200 chr or Whatman 17 chr); and (3) two types of funnel (circular or rectangular) at five synthetic sludge concentrations (g/l)

Sludge g/l	Paper	Funnel	CST (s) without sealant			CST (s) with sealant		
			N	Mean	CV%	N	Mean	CV%
2.3	Fisher	Circular	4	6.0	5.5	9	8.6	4.5
2.3	Fisher	Rect.	14	7.1	7.9	14	7.0	6.7
2.3	Whatman	Circular	12	11.7	3.4	8	12.2	2.5
2.3	Whatman	Rect.	12	9.6	8.1	15	11.4	6.5
5.64	Fisher	Circular	20	40.8	16.7	9	51.6	11.5
5.64	Fisher	Rect.	24	29.5	15.8	7	40.9	13.6
5.64	Whatman	Circular	10	49.2	22.2	10	66.2	4.0
5.64	Whatman	Rect.	10	43.7	14.1	7	43.4	8.5
8.8	Fisher	Circular	9	65.2	32.5	10	127.7	14.8
8.8	Fisher	Rect.	3	30.3	10.2	10	82.9	9.3
8.8	Whatman	Circular	5	74.1	20.3	8	177.1	15.0
8.8	Whatman	Rect.	12	62.7	16.7	10	72.6	9.3
12.1	Fisher	Circular	13	161.3	24.5	6	162.9	13.4
12.1	Fisher	Rect.	3	158.7	8.1	3	196.5	7.0
12.1	Whatman	Circular	8	179.8	17.2	6	397.4	12.6
12.1	Whatman	Rect.	5	102.1	9.9	4	272.4	9.7
15.3	Fisher	Circular	3	240.9	26.9	-	-	-
15.3	Fisher	Rect.	3	184.1	24.3	3	229.7	19.1
15.3	Whatman	Circular	5	359.1	7.1	4	895.5	5.9
15.3	Whatman	Rect.	5	228.5	7.2	3	364.1	6.2

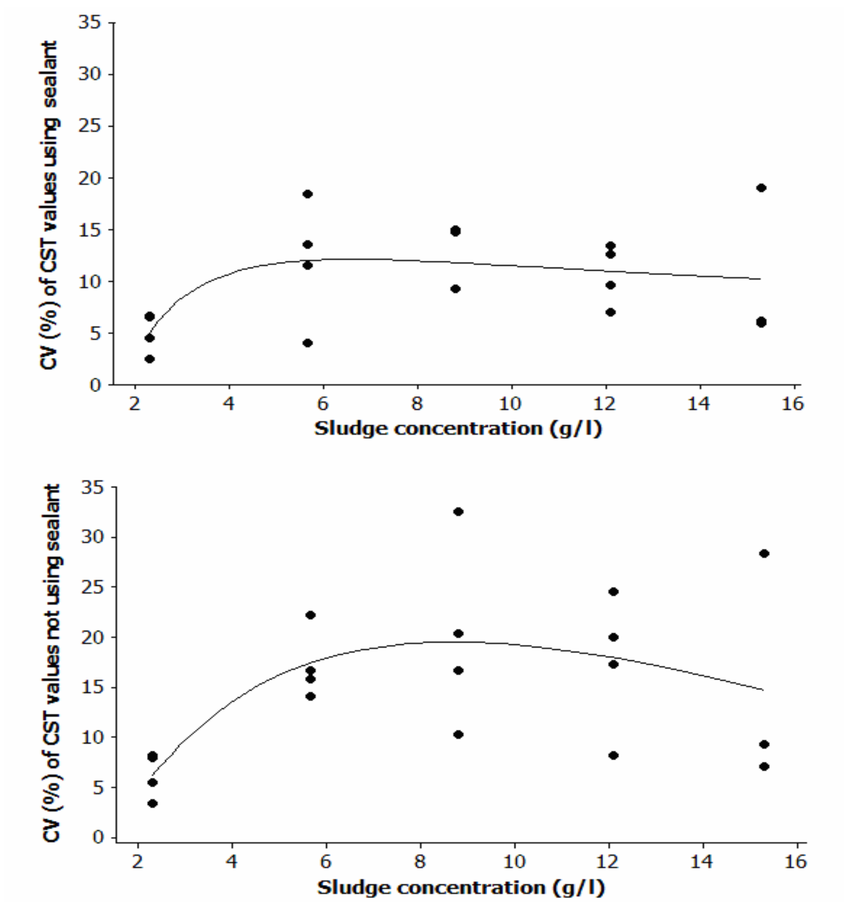
CV% coefficient of variation; Rect. rectangular; - missing value

The mean CST estimates were generally higher when (a) the estimates were made with the sealant compared to without the sealant; (b) when Whatman 17 chr filter papers were used compared to when Fisher 200 chr filter papers were used; and (c) when circular funnels were used compared with when rectangular funnels were used.

There was considerable variability within and between the estimates of CST. The pattern of variability was that the coefficients of variation were higher when no sealant was used (3.4% to 32.5%) compared to when there was sealant (2.5% to 19.1%).

The relationships between the coefficients of variation of the CST values and the sludge concentrations were observed to be non-linear (Figure 4.4.1). However, $\log_e$ transformations were found to make these relationships linear (Figures 4.4.2 to 4.4.5). Logarithmic transformations of both CST and sludge concentrations are necessary to homogenize the variances and normalize the residuals, and thereby eliminate violations of the assumptions of regression analysis.

Figure 4.4.1 Relationships between the coefficients of variation in the CST values and the sludge concentrations with and without the use of the sealant



The values of $\log_e$ CST were regressed on the $\log_e$ sludge concentrations (the covariate) for each combination of the three experimental factors. When the factors were partitioned with respect to sealant, filter paper, and funnels, the R^2 values obtained using simple linear regression equations indicated that a very high proportion (92.1% to 99.1%) of the variation in the CST values could be explained by the variations in the sludge concentrations. The F statistics indicated that all the regression models were significant at the 0.01 level, whilst the t statistics indicated that the intercepts and slopes of all the equations were significantly different from zero at the 0.01 level (Tables 4.4.2, 4.4.4, 4.4.6, and 4.4.8).

Figure 4.4.2 Linear regression of log CST versus log sludge concentration using Fisher 200 chr papers and circular funnels

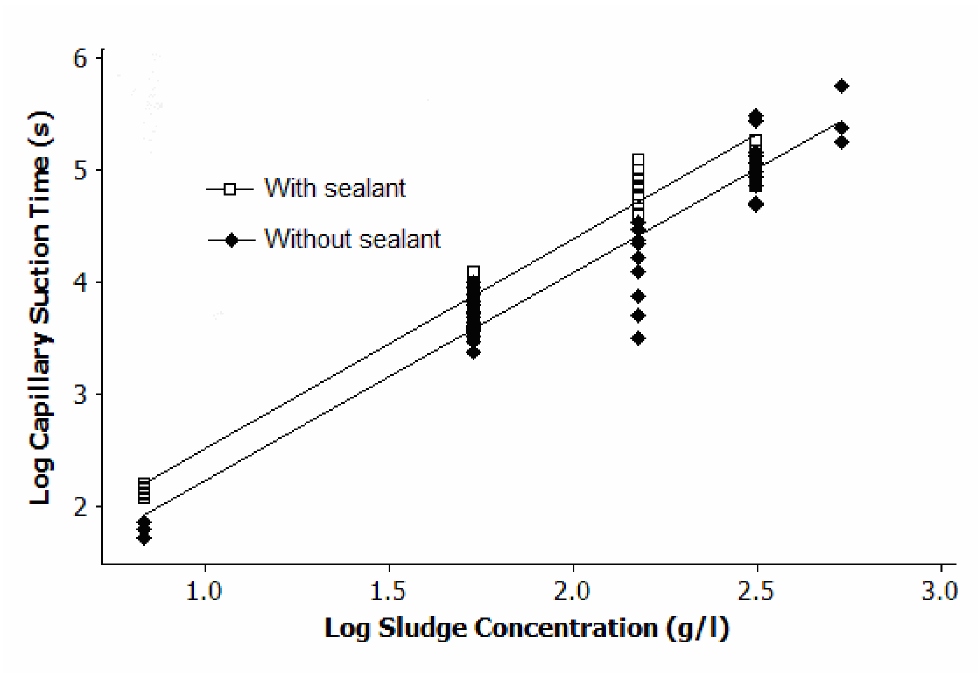


Table 4.4.2 Linear regression of log CST versus log sludge concentration using Fisher 200 chr papers and circular funnels

Treatment	Regression coefficients		t statisti	R^2 %
Without sealant	Intercept	0.3081	2.34*	92.1%
	Slope	1.8523	23.48*	
With sealant	Intercept	0.6456	7.28*	98.0%
	Slope	1.8694	39.21*	

* Significant at $\alpha = 0.01$

Table 4.4.3 Testing the equality of intercepts and slopes the regression lines in Figure 4.4.2

Null hypothesis	F statistic	P
No difference between intercepts	2.01	0.161
No difference between slopes	0.03	0.858

Figure 4.4.3 Linear regression of log CST versus log sludge concentration using Whatman 17 chr papers and circular funnels

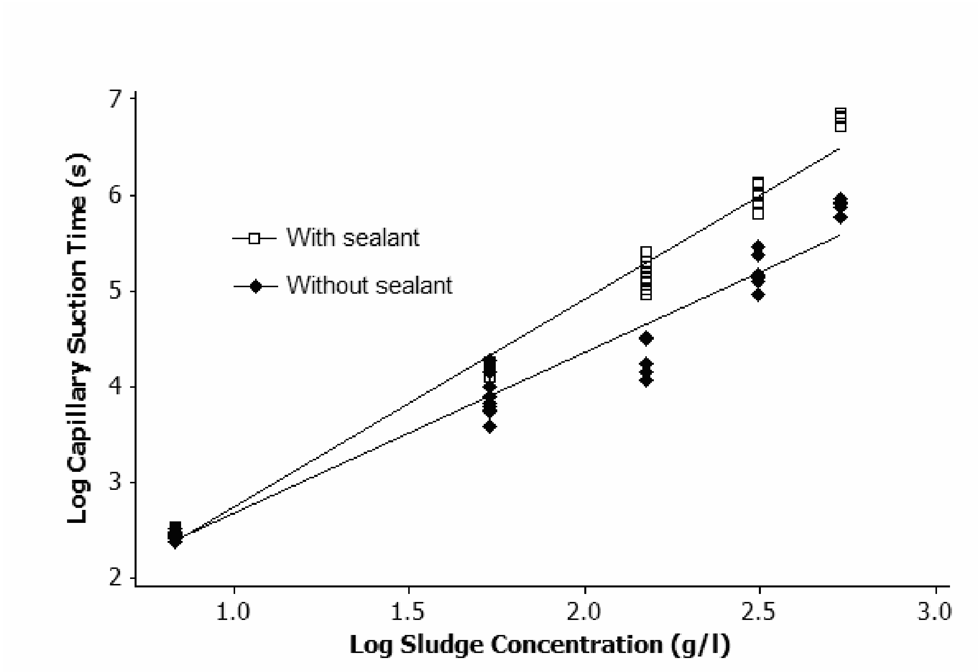


Table 4.4.4 Linear regression of log CST versus log sludge concentration using Whatman 17 chr papers and circular funnels

Treatment	Regression coefficients		t statistic	R^2 %
Without sealant	Intercept	1.0002	10.30*	96.7 %
	Slope	1.6798	33.36*	
With sealant	Intercept	0.5744	6.38*	98.5 %
	Slope	2.1658	47.52*	

* Significant at α = 0.01

Table 4.4.5 Testing the equality of intercepts and slopes the regression lines in Figure 4.4.3

Null hypothesis	F statistic	P
No difference between intercepts	3043.3	0.000*
No difference between slopes	48.6	0.000*

*Significant at α = 0.01

Figure 4.4.4 Linear egression of log CST versus log sludge concentration using Fisher 200 chr papers and rectangular funnels

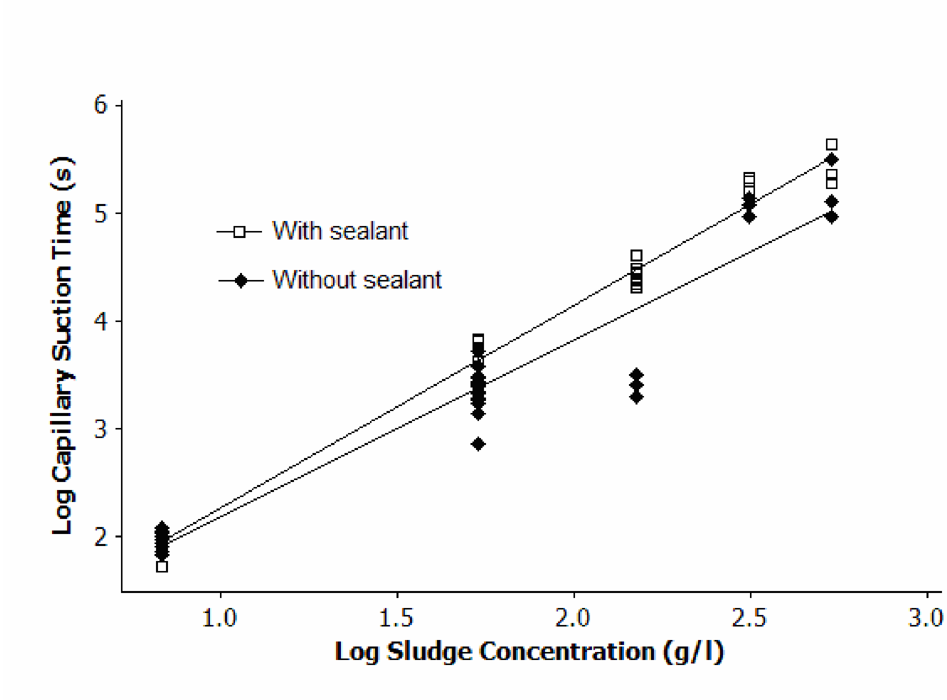


Table 4.4.6 Linear regression of log CST versus log sludge concentration using Fisher papers and rectangular funnels

Treatment	Regression coefficients		t statistic	R^2 %
Without sealant	Intercept	0.5394	4.84*	93.4%
	Slope	1.6438	25.17*	
With sealant	Intercept	0.3837	7.15*	99.1%
	Slope	1.8827	62.93*	

* Significant at α = 0.01

Table 4.4.7 Testing the equality of intercepts and slopes the regression lines in Figure 4.4.4

Null hypothesis	F statistic	P
No difference between intercepts	2315.48	0.000*
No difference between slopes	10.62	0.002*

* Significant at α = 0.01

Figure 4.4.5 Linear regression of log CST versus log sludge concentration using Whatman 17 chr papers and rectangular funnels

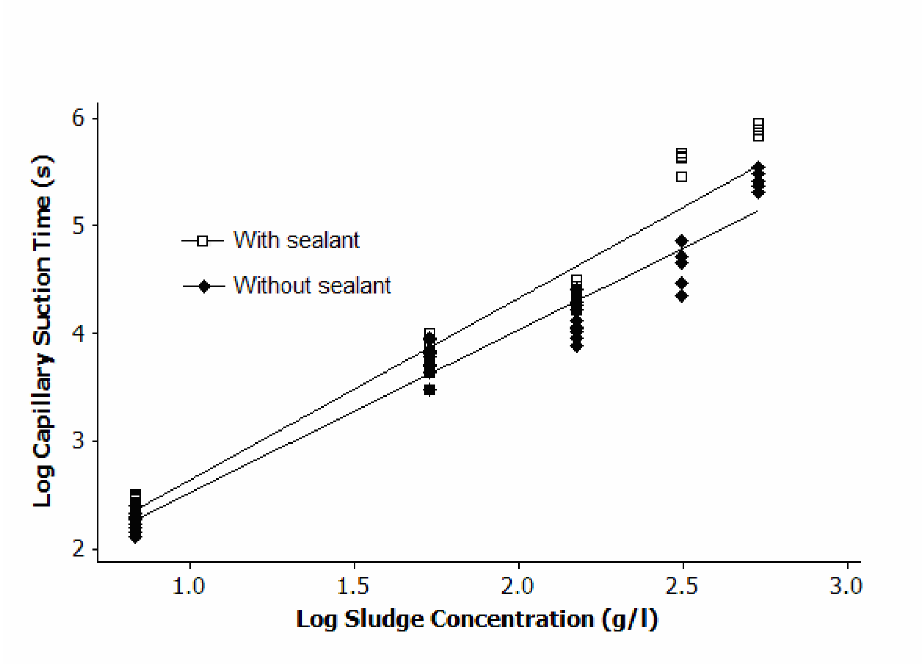


Table 4.4.8 Linear regression of log CST versus log sludge concentration using Whatman 17 chr papers and rectangular funnels

Treatment	Regression coefficients		t statistic	R^2 %
Without sealant	Intercept	1.0023	10.84*	96.0%
	Slope	1.5141	31.56*	
With sealant	Intercept	0.9433	8.78*	95.6%
	Slope	1.6949	28.32*	

* Significant at $\alpha = 0.01$

Table 4.4.9 Testing the equality of intercepts and slopes the regression lines in Figure 4.4.5

Null hypothesis	F statistic	P
No difference between intercepts	1778.2	0.000*
No difference between slopes	5.64	0.020*

Significant at $\alpha = 0.01$

To determine the effects of sealant on the results of the CST tests, the coefficients of pairs of linear regression lines were compared. The null hypotheses were that the slopes and the intercepts of the regression lines derived from the estimates of CST obtained with the sealant (using various combinations of filter papers and funnels) were not significantly different from the slopes and the intercepts of the regression lines derived from the CST estimates obtained without the sealant (using the same combinations of filter papers and funnels). The models assumed that $\log_e$ sludge concentration, which was linearly related to $\log_e$ CST, was a covariate i.e., held statistically constant, so that only the effects of the sealant were analyzed for each combination of filter papers and funnels.

The results comparing the coefficients of the regression lines are presented in Tables 4.4.3, 4.4.5, 4.4.7, and 4.4.9. For all but one of the combination of factors (Fisher 200 chr paper and circular funnel in Table 4.4.3) there was a significant difference at the 0.01 level between the slopes and the intercepts of the regression lines derived from the estimates of CST obtained with the sealant (at different combinations of filter papers and funnels) and the slopes and the intercepts of the regression lines derived from the estimates obtained without the sealant (at the same combinations of filter papers and funnels).

The slopes of the regression lines extracted from the data using the sealant were consistently higher than the slopes extracted from the data obtained when the sealant was not used (Tables 4.4.2, 4.4.4, 4.4.6 and 4.4.8). This indicated that the increases in the CST values per unit increase in sludge concentration were consistently higher when sealant was used, compared to when the sealant was not used.

The regression models were applied to predict the mean CST values at a known fixed sludge concentration for comparison with the results of the CST tests obtained experimentally. The eight regression equations in Tables 4.4.2, 4.4.4, 4.4.6 and 4.4.8 were each interpolated with a sludge concentration of 10.0 g/l (specifically, a $\log_e$ sludge concentration of 2.30259) in order to predict the CST values at the eight combinations of the three factors. 10.0 g/l was chosen as the predictor because it was in the middle of the range of sludge concentrations used experimentally for the CST tests.

The predictions provided a simplified summary of the results obtained experimentally. The mean CST values ± 95% confidence intervals predicted by the regression models using a sludge concentration of 10 g/l (Table 4.4.10) were within the ranges of CST test results for sludge concentrations between 8.8 and 12.1 g/l obtained experimentally (Table 4.4.1).

Table 4.4.10 Predicted mean CST values ± 95% confidence limits at a sludge concentration of 10 g/l computed by use of regression equations

		With sealant		Without sealant	
Paper	Funnel	Predicted Mean CST (s)	Predicted 95% Confidence Intervals	Predicted Mean CST (s)	Predicted 95% Confidence Intervals
Whatman	Circular	260.2	242.2 – 279.5	130.1	119.0 – 142.2
Fisher	Circular	141.2	130.4 – 152.9	104.1	94.9 - 114.1
Whatman	Rectangular	127.2	113.3 – 142.8	89.0	82.1 – 96.5
Fisher	Rectangular	112.0	105.7 – 118.7	75.6	67.1 – 85.2

The highest mean CST values (112.0 s to 260.2 s) were predicted when the sealant was used, whereas the lowest predicted mean CST values (75.6 s to 130.1 s) were predicted when there was no sealant (Table 4.4.10). The prediction of the highest mean CST value (260.2 s) using the sealant was for the Whatman 17 chr paper and the circular funnel. The lowest predicted mean CST value (112.0 s) using the sealant was for the Fisher 200 chr paper and the rectangular funnel. Without the sealant, the highest predicted mean CST was also for the Whatman 17 chr paper and circular funnel (130.1 s) whilst the lowest prediction of mean CST was for the Fisher 200 chr paper and the rectangular funnel (75.6 s).

To quantify the amount of leakage further experiments were carried out. The volumes of the synthetic sludge that disappeared within 1 min was recorded for Whatman 17 chr and Fisher 200 chr filter papers (with and with out using the sealant), and the results are summarized in Table 4.4.11. For the filter papers, the volume of sludge sample that disappeared was higher for the synthetic sludge without using the sealant. The reductions in the amount of liquid that disappeared when the sealant was used was in the range of 0.2 for Whatman 17 chr and 0.1 for Fisher 200 chr. These reductions were significant ($p<0.05$), and they prove that there is significant leakage occurring between the funnel and the filter paper leading to unwanted increases in the variability of the CST test data. Using the sealant prevented or at least reduced leakages, which improves and minimizes the variability of the CST tests.

Table 4.4.11 The mean volume of the leakage after 1 min with and without using the funnel sealant for Whatman 17 chr and Fisher 200 chr papers

Filter paper	Sealant		
	with	Without	Volume of the leakage (ml)
Whatman 17 chr	0.2^{b}	0.4^{a}	0.2
Fisher 200 chr	0.3^{b}	0.4^{a}	0.1

$^{a, b}$ Different superscripts for the same filter paper and the volume with and without the sealant are significantly different ($p < 0.05$).

4.4.4 Discussion

The use of a sealant influenced the variability in the results of the CST tests. The coefficients of variation were higher when no sealant was used (3.4% to 32.5%) compared to when there was sealant (2.5% to 19.1%). The coefficients of variation varied non-linearly with respect the sludge concentrations, and could not be predicted using regression models.

The highest mean CST values were observed when the sealant was used, whereas the lowest mean CST values were obtained when there was no sealant. When the sealant was used, the highest mean CST values were obtained using Whatman 17 chr paper and a circular funnel, compared with the lower values obtained using Fisher 200 chr paper and a rectangular funnel. When the sealant was not used, the highest mean CST estimates were also observed using Whatman 17 chr paper and a circular funnel, compared with the lower values obtained using Fisher 200 chr paper and a rectangular funnel.

The results confirmed the hypothesis that reducing the leaks between the funnel and the paper would improve the results of CST tests, and lower the variability between replicate tests. The differences between the CST test results obtained using Whatman 17 chr with circular funnels compared with Fisher 200 chr papers and rectangular funnels reflecting interactions between paper type and funnel type.

The slopes of the linear regression lines of CST on sludge concentration (i.e., the rates at which the CST values increased per unit increase in sludge concentration) were consistently higher when the sealant was used, compared to when the sealant was not used. It shows that the filtrate leakage occurred at all sludge concentrations at similar rate when the sealant was not used. It may be inferred that the sealant was effective in reducing the unwanted filtrate leakage at different concentration levels.

There was a noticeable decrease in the filtrate leakage when the sealant was used for both Whatman 17 chr and Fisher 200 chr filter papers. Viscosity is known to affect the movement of filtrate through porous material (Forster, 2002; Abu-Orf and Örmeci, 2005). The amount of leakage of filtrate between paper and funnel is expected to be affected by the properties of the tested suspension. This was shown to have an effect when testing the effect of sealant at different sludge concentrations (different slopes of the regression of log CST on log sludge concentration).

The CST test assumes that all filtrate is constrained within the paper. Preventing leakage between the funnel and the paper is therefore essential to reduce data variability. The leakage that occurs during testing without a sealant is random in nature. Besides other variables, it also depends on how well the funnel is attached to the device (e.g. flatness and uniformity of the surface of the paper). The hypothesis is that when leakage is prevented, the CST becomes more consistent (due to control over random leakage). Preventing leakage is expected to increase the mean CST value, which was actually observed in this study.

These results are not comparable with those of any other researchers, since the literature review revealed no previous studies on the effects of using a sealant on the results of CST tests.

Chapter Five

Modelling the effects of temperature on CST results♣

5.1 Introduction

Three experiments were performed with the aim of exploring the influence of temperature on the results of CST tests, using different synthetic sludge concentrations, formulations, filter papers, and funnels.

5.2 Experimental design

The objective of the first experiment was to obtain data which could be used to model the relationships between temperature and the results of CST tests. A replicated multi-factorial experimental design was used to study the effects of two main factors on the results of the CST tests. These factors were (1) temperature, with four levels at 10°C, 15°C, 20°C, 25°C covering the normal temperature range in the UK and (2) synthetic sludge concentration, with six levels of total suspended solids (TSS) at 2.3, 5.64, 8.8, 12.1, 15.3, and 31.6 g/l. A total of 479 estimates of CST were

♣ The content of this chapter has been submitted as a manuscript to the Journal of Environmental Engineering.

Sawalha, O., and Scholz, M. 2010. Impact of temperature on the capillary suction time. *Journal of Environmental Engineering-ASCE*, submitted.

made, with between 5 and 10 replicate measurements for each combination of factors (Table 1, Appendix). All CST estimates were made using Whatman 17 chr papers or Fisher 200 chr papers, with circular or rectangular funnels, but with no stirring, and no sealant. The results of the CST tests (dependent variables) were modelled using ANCOVA and MLR. The TSS (total suspended solid concentration of synthetic sludge) temperature, paper type (Fisher 200 chr or Whatman 17 chr), and funnel geometry (circular or rectangular) were the independent variables. To determine which transformations of the variables would create the best fitting models, Box-Cox test was used. A logarithmic transformation of the dependent variable was justified since the optimum Box-Cox transformation parameter for CST was $\lambda = 0.0$. Dummy binary variables were included to represent the paper types and funnel types where 1 = Whatman 17 chr paper; 0 = Fisher 200 chr paper; 0 = circular funnel; 1 = rectangular funnel.

The objective of the second experiment was to investigate the relationship between sludge desorptivity, the results of CST tests, and temperature. The desorptivity of synthetic sludge samples was estimated at sludge TSS concentrations of 2.3, 5.64, 8.8, 12.1, 15.3 and 31.6 g/l and temperatures of 10, 15, 20, and 25^{o}C using standard CST test apparatus (Whatman 17 chr filter paper and circular funnel).

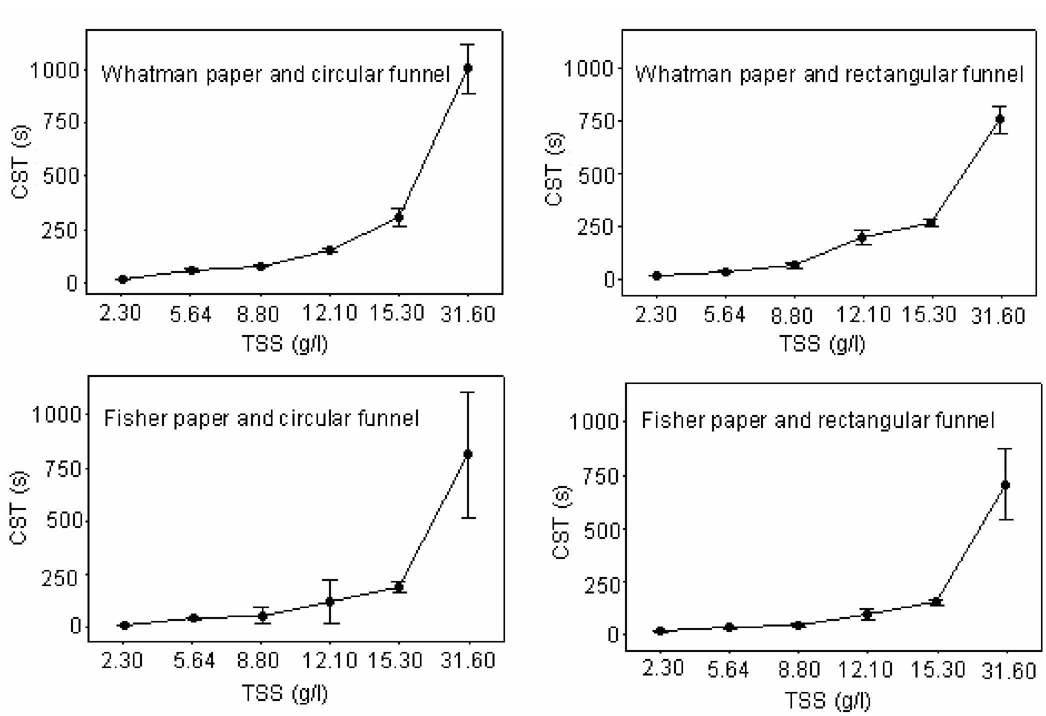
The objective of the third experiment was to examine the relationships between the CST, the chemical composition of sludge and the temperature. The CST of simplified synthetic sludges was estimated using the standard apparatus (Whatman 17 chr paper and circular funnel) when each ingredient was added in sequence, one at a time (to form eight different sludge formulations), at temperatures of 10, 15, 20, 25, and 30°C. This experiment enabled the CST behaviour of each ingredient to be explored with respect to changes in temperature.

5.3 Results

5.3.1 Modelling the relationship between CST and temperature

The relationships between the mean CST estimates and the synthetic sludge TSS concentrations at 15°C are visualized in Figure 5.3.1. The mean CST values increased non-linearly with the sludge concentrations between 2.3 g/l and 31.6 g/l using Whatman 17 chr and Fisher 200 chr papers with circular and rectangular funnels. Thus, the TSS had to be taken into account when considering the relationship between the results of CST tests and temperature.

Figure 5.3.1 Estimates of mean CST (s) ± 95% confidence intervals at 15°C



The relationships between the mean CST values and the temperatures at each sludge concentration were generally approximated by “U” or “V” shaped curves. The mean CST values generally declined between 10-20°C, then mostly increased between 20-25°C (Figure 5.3.2). The coefficients of variation in the estimates of CST were erratic, ranging 2.4% to 33.2%. No general patterns amongst the coefficients of variation in the estimates of CST were identified when the results of CST tests between Whatman 17 chr papers (Table 5.3.1) and Fisher 200 chr papers were compared (Table 5.3.2). The coefficients of variation did not increase or decrease systematically with respect to the sludge concentrations or the temperatures.

Figure 5.3.2 Variations in the mean CST (s) with respect to synthetic sludge TSS concentration (g/l) and temperature (°C) using circular and rectangular funnels for (a) Whatman 17 chr filter papers and (b) Fisher 200 chr papers

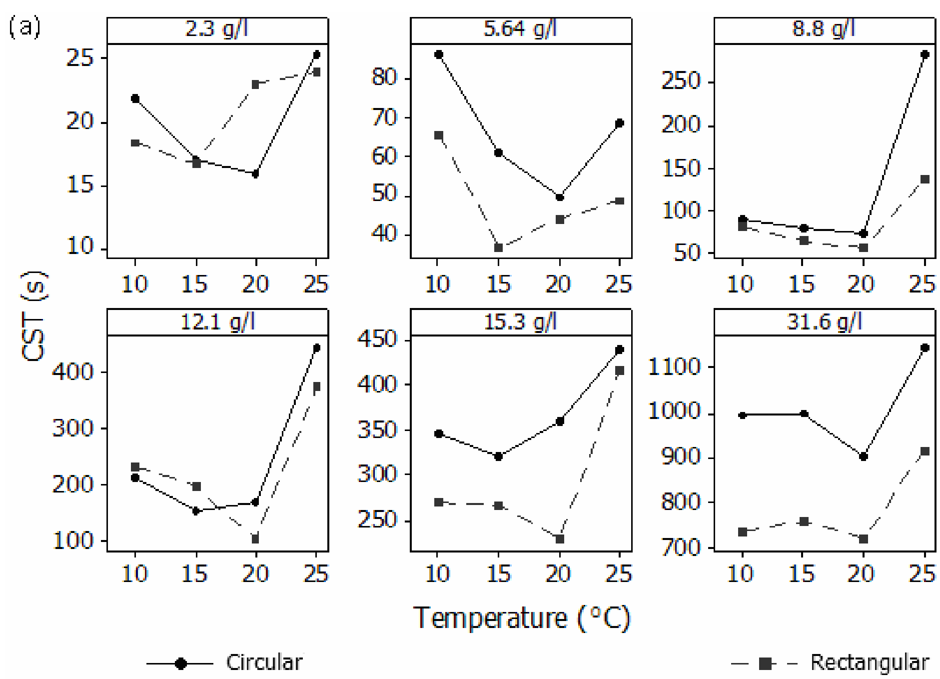


Figure 5.3.2 continued

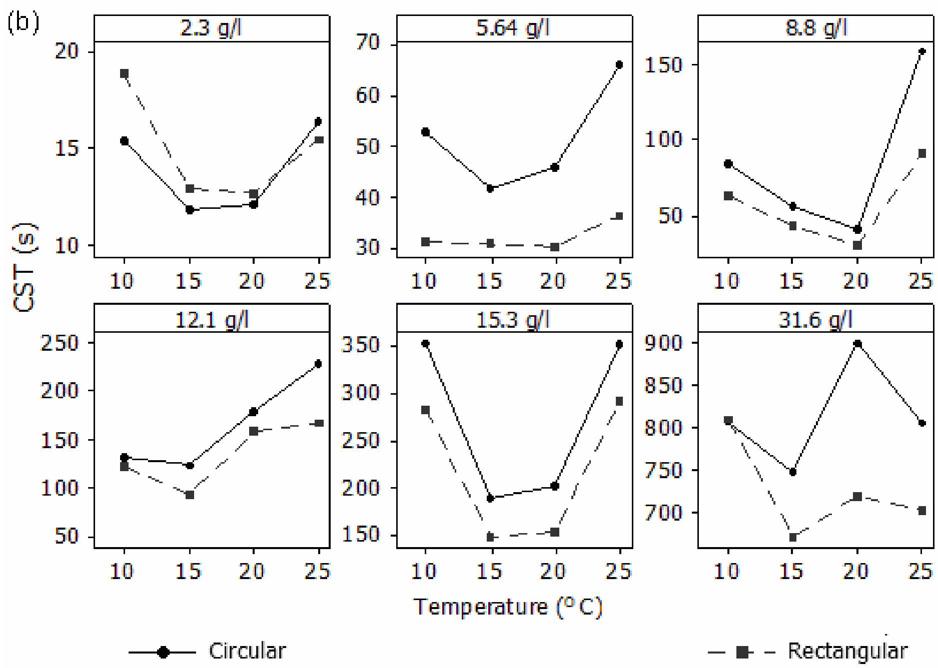


Table 5.3.1 Coefficients of variation of CST estimates with respect to synthetic sludge TSS concentrations (g/l) and temperatures (°C) using Whatman 17 chr papers and circular funnel

Temperature °C	Sludge concentration TSS (g/l)					
	2.30	5.64	8.80	12.1	15.3	31.6
	Coefficient of variation of CST (%)					
10	3.06	14.27	4.95	31.55	8.32	14.74
15	5.96	12.97	5.05	4.95	10.99	7.31
20	10.41	21.06	20.27	3.26	7.06	4.99
25	7.43	14.89	7.55	7.68	10.56	9.50

Table 5.3.2 Coefficients of variation of CST estimates with respect to synthetic sludge TSS concentrations (g/l) and temperatures (°C) using Fisher 200 chr papers and circular funnel

Temperature °C	Sludge concentration TSS (g/l)					
	2.30	5.64	8.80	12.1	15.3	31.6
	Coefficient of variation of CST (%)					
10	27.02	22.85	18.89	2.35	15.22	11.35
15	8.27	12.53	27.45	33.23	6.18	14.55
20	13.29	12.15	18.27	21.56	26.93	8.28
25	3.66	15.48	19.66	19.08	6.00	2.42

The non-linear relationships between the mean estimates of CST, the temperatures, the synthetic sludge TSS concentrations, the paper types and the funnel types are visualized in bar charts with 95% confidence intervals (Figures 5.3.3 and 5.3.4). If CST varied with temperature following the Arrhenius model, then the relationships between the logarithms of CST and the reciprocals of temperature should theoretically be linear. Visual evidence is provided to indicate that the relationships between the log mean CST estimates and 1/temperature were not linear using Whatman 17 chr papers (Figure 5.3.5) or Fisher 200 chr papers (Figure 5.3.6) with circular and rectangular funnels at sludge concentrations from 2.3 g/l to 31.6 g/l at temperatures between 15^{o}C and 25^{o}C. Linear relationships were visualized between the log CST values and the log sludge concentrations at the four temperatures when the CST estimates using different types of funnel and filter were pooled (Figure 5.3.7). These linear relationships implied that the logarithms of the sludge concentrations could be incorporated as a covariate in an ANCOVA model to determine the effects of temperature on the results of CST tests.

Figure 5.3.3 Relationships between mean CST (s) ± 95% confidence intervals, TSS (g/l), temperature (^{o}C) using Whatman 17 chr filter paper and circular funnel

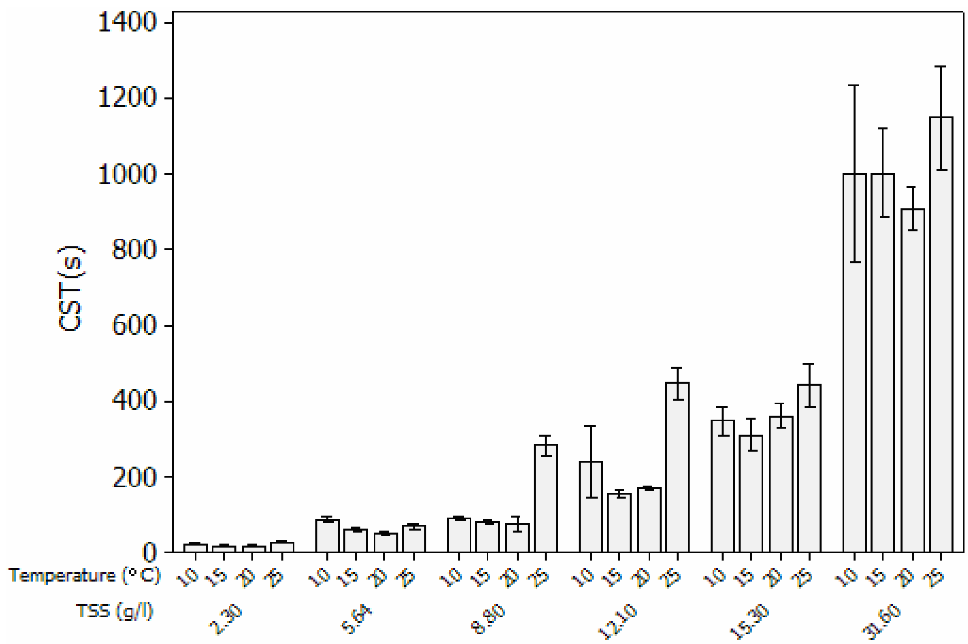


Figure 5.3.4 Relationships between mean CST (s) ± 95% confidence intervals, TSS (g/l), and temperature (^{o}C) using Fisher 200 chr filter paper and circular funnel

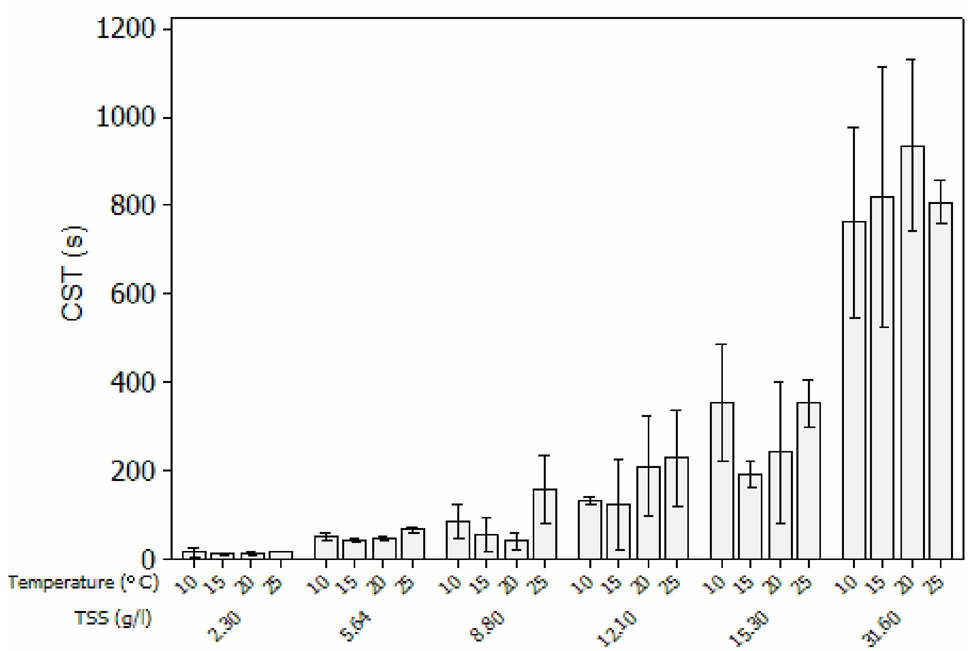


Figure 5.3.5 Relationships between log mean CST (s) and 1/temperature (^oC) using Whatman 17 chr papers, circular and rectangular funnels

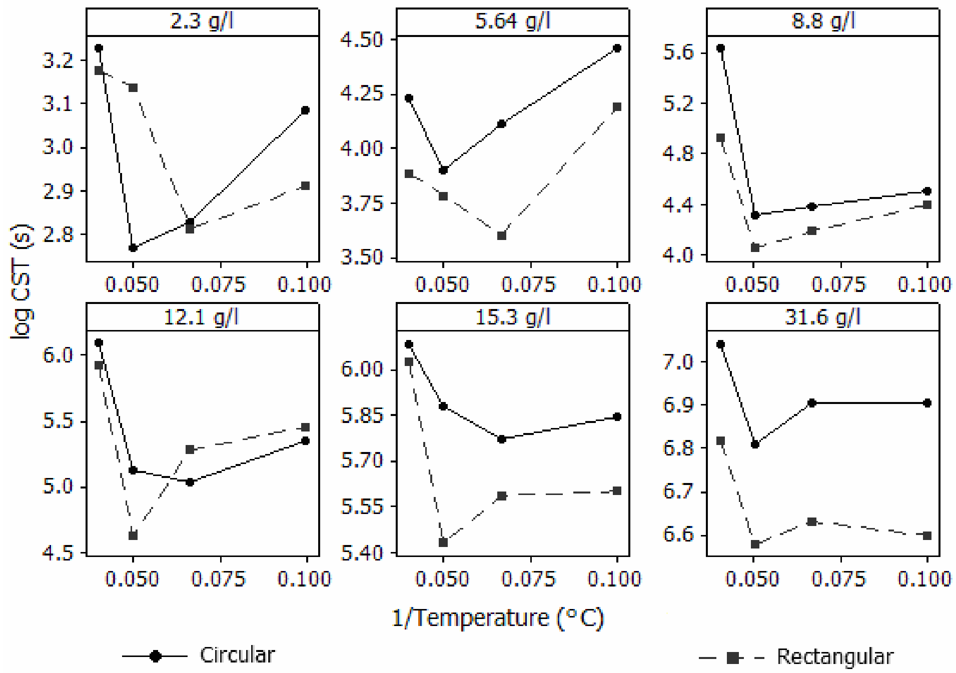


Figure 5.3.6 Relationships between log mean CST (s) and 1/temperature (^oC) using Fisher 200 chr papers, circular and rectangular funnels.

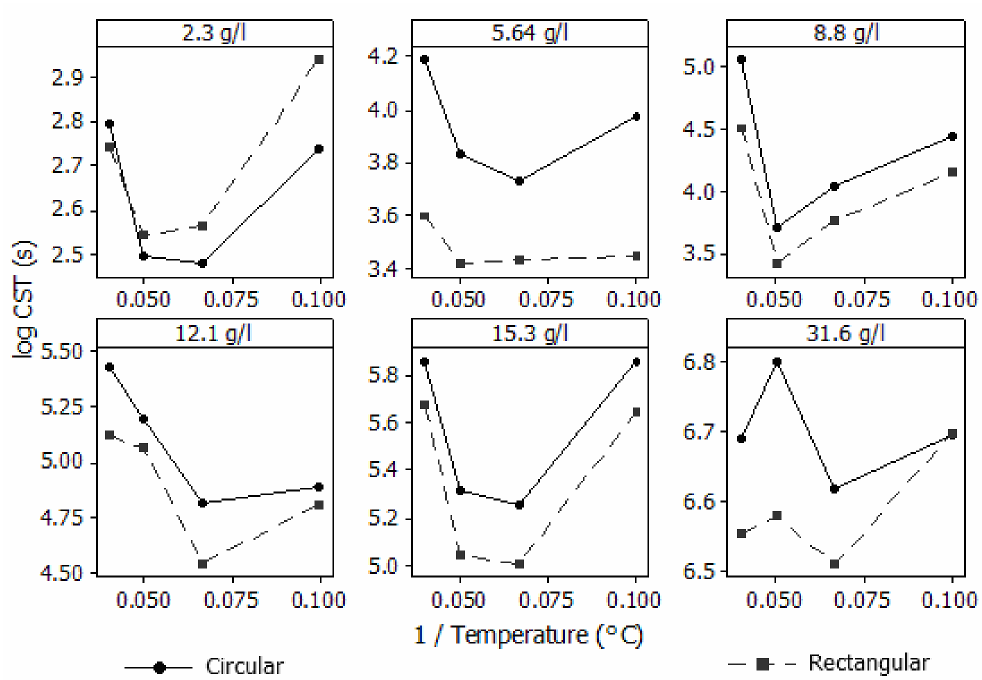


Figure 5.3.7 Relationships between log CST (s) and log sludge TSS (g/l) with respect to temperature (^oC)

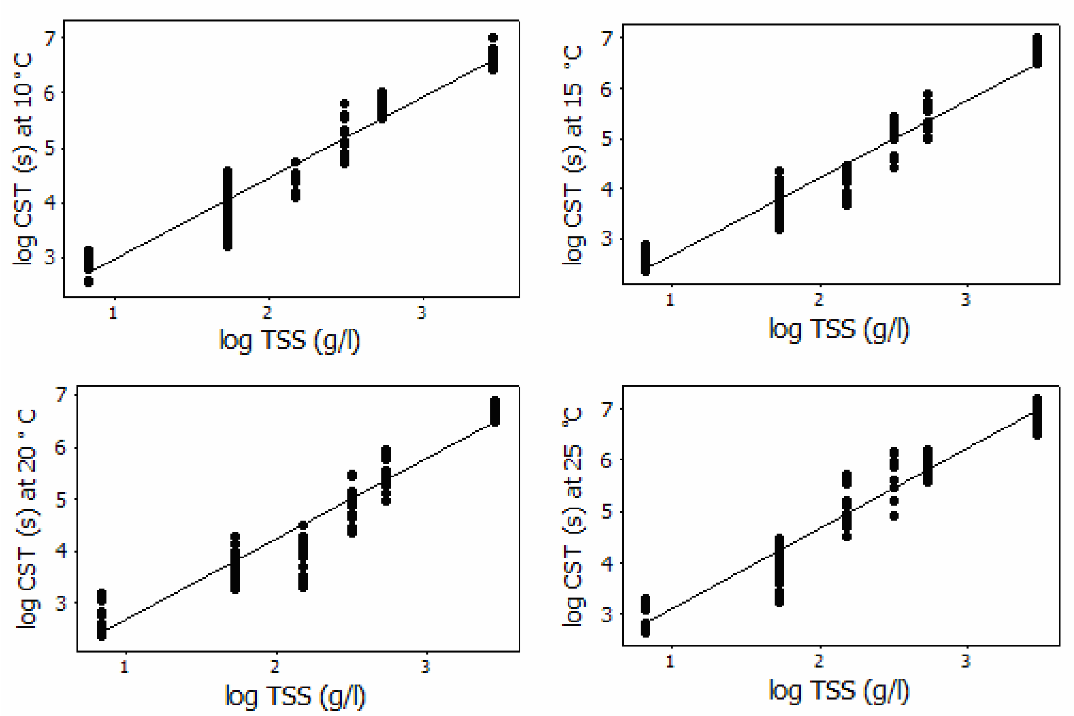
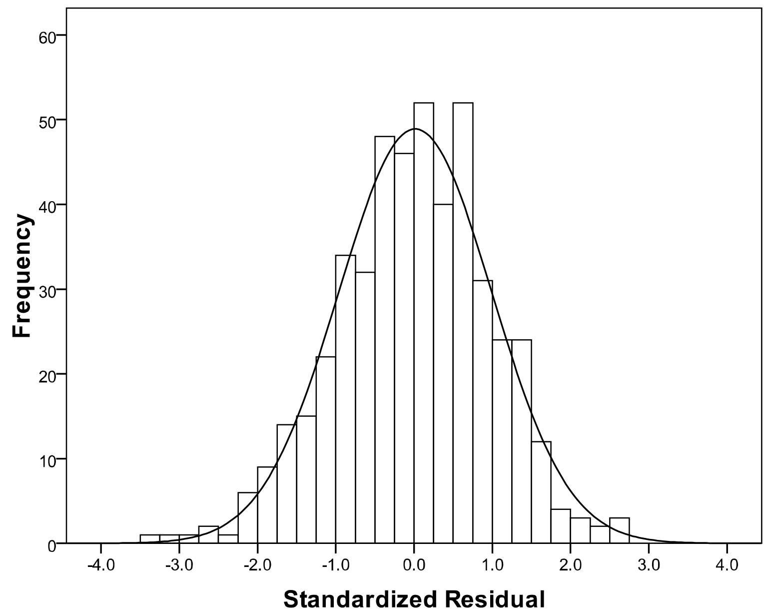


Table 5.3.3 Multi-factorial ANCOVA (with log TSS as a covariate) to compare the mean CST estimates with respect to temperature

Source	Sum of Squares	Degrees of Freedom	Mean Square	F statistic	p
Log TSS (covariate)	643.201	1	643.201	7450.44	0.000*
Temperature	15.105	3	5.035	58.32	0.000*
Paper (Whatman or Fisher)	12.426	1	12.426	143.94	0.000*
Funnel (Circular or Rectangular)	6.886	1	6.886	79.76	0.000*
Temperature x Paper	0.553	3	0.184	2.14	0.095
Temperature x Funnel	0.275	3	0.092	1.06	0.364
Paper x Funnel	0.131	1	0.131	1.52	0.218
Temperature x Paper x Funnel	0.027	3	0.009	0.11	0.957
Error	39.885	462	0.086		
Total	10815.15	479			

* Significant at $\alpha = 0.01$

Figure 5.3.8 Normal distribution of residuals for the ANCOVA model



No threats to the validity of the results of ANCOVA caused by violations of the theoretical assumptions were found. Levene's test indicated that the variances were homogeneous, and the Anderson-Darling test revealed that the residuals did not deviate from normality at the 0.01 level (Figure 5.3.8). The results of ANCOVA indicated that the mean CST varied significantly at $\alpha = 0.01$ between temperatures of 10^{o}C to 25^{o}C (Table 5.3.3) when TSS was included as a covariate, and thereby held statistically constant. Both the paper type and funnel geometry also had a significant effect on the mean CST at $\alpha = 0.01$; however, no significant interaction was found between temperature, paper type, and funnel geometry. No interaction implied that the effects of temperature on the CST were the same when using Whatman 17 chr and Fisher 200 chr papers and when using circular and rectangular funnels. ANCOVA provided evidence to conclude that the effect of temperature on the CST was not influenced by the filter paper type or by the funnel geometry. The effects of temperature on the results of the CST tests were independent of the apparatus used.

The relationships between the log CST estimates (dependent variable) and four independent variables (temperature, TSS, paper type, and funnel type) were modelled using multiple linear regression analysis (MLR). The stepwise MLR procedure was applied to determine an optimum subset of independent variables that could be used to predict the mean values of log CST.

Two models were extracted from the data. Model I (Table 5.3.4) used polynomial terms with squared transformations to fit non-linear relationships between log CST, TSS, and temperature than logarithmic transformations. Model I was defined by equation 5.3.1.

$$\log CST = 3.869 + 0.301\ TSS - 0.005\ TSS^2 - 0.226\ \text{temperature} + 0.007\ \text{temperature}^2 + 0.298\ \text{paper} - 0.241\ \text{funnel} \qquad (5.3.1)$$

where CST = capillary suction time (s); TSS = total suspended solids (g/l); temperature = ^{o}C; Fisher 200 chr paper = 0; Whatman 17 chr paper = 1; circular funnel = 0; rectangular funnel = 1.

The R^2 statistic indicated that a substantive proportion (95.7%) of the variability in the dependent variable was explained. The value of F statistic indicated a significant amount of variability was explained at the 0.01 level. The t tests indicated that the intercept and partial regression coefficients were significantly different from zero at $\alpha = 0.01$. There were no threats to the validity of the model caused by violations of theoretical assumptions. The residuals were randomly and evenly scattered either side of their mean (zero) value (Figure 5.3.9) indicating that the variances were homogeneous. The frequency distribution histogram (Figure 5.3.10) and the results of the Anderson-Darling test provided evidence to indicate that the residuals were normally distributed.

Table 5.3.4 MLR Model I to predict log CST using TSS, TSS2, temperature, temperature2, paper type, and funnel type as the independent variables

(a) ANOVA and R^2

Source of variance	Sum of Squares	Degrees of Freedom	Mean Square	F statistic	P	Adjusted R^2 (%)	Standard error of estimate
Regression	695.209	6	115.868	1756.362	0.000*	95.7	0.256
Residual	31.138	472	0.066				
Total	726.347	478					

(b) Regression coefficients

Variable	Regression Coefficients	t statistic	p	95% confidence intervals of regression coefficients	
Intercept	3.869	27.732	0.000*	3.595	4.143
TSS	0.301	54.074	0.000*	0.290	0.312
TSS^2	-0.005	-32.521	0.000*	-0.005	-0.005
Temperature	-0.226	-13.619	0.000*	-0.258	-0.193
Temperature ^{2}	0.007	14.466	0.000*	0.006	0.008
Paper	0.298	12.491	0.000*	0.251	0.340
Funnel	-0.241	-10.253	0.000*	-0.287	-0.195

* Significant at $\alpha = 0.01$

Table 5.3.5 MLR Model II to predict log CST using log TSS, log temperature, paper type, and funnel type as the independent variables

(a) ANOVA and R^2

Source of variance	Sum of Squares	Degrees of Freedom	Mean Square	F statistic	P	Adjusted R^2 (%)	Standard error of estimate
Regression	670.242	4	167.560	1415.622	0.000*	92.2%	0.344
Residual	56.105	474	0.118				
Total	726.347	478					

(b) Regression coefficients

Variable	Regression Coefficients	t	P	95% confidence intervals of regression coefficients	
Intercept	0.877	6.323	0.000*	0.604	1.149
log TSS	1.536	73.734	0.000*	1.495	1.577
log Temperature	0.134	2.914	0.004*	0.044	0.224
Paper	0.326	10.211	0.000*	0.263	0.388
Funnel	-0.237	-7.526	0.000*	-0.298	-0.175

* Significant at $\alpha = 0.01$

Model II (Table 5.3.5) used logarithmic transformations to fit the non-linear relationships between log CST, TSS. Model II was defined by equation 5.3.2.

$$\log CST = 0.877 + 1.536 \log TSS + 0.134 \log temperature + 0.326\ Paper - 0.237\ Funnel \quad (5.3.2)$$

The R^2 statistic indicated that Model II explained a smaller proportion (92.2%) of the variability in the dependent variable than Model I. The standard error of Model II (0.344) was also higher than Model I (0.256) indicating that the predictions of Model II were not as precise as those of Model I. The value of the F statistic of the model indicated a significant amount of variability was explained at the 0.01 level. The t tests indicated that the intercept and partial regression coefficients were significantly different from zero at $\alpha = 0.01$. There was a threat to the validity of Model II caused by violations of theoretical assumptions. Visual examination of the residual plots revealed that the residuals were clustered into three groups, and were not randomly or evenly scattered either side of their mean (zero) value (Figure 5.3.11) indicating that the variances were not homogeneous. In comparison, the residuals for Model I were relatively more evenly and randomly distributed either side of the mean (Figure 5.3.9). For this reason, Model I was considered to be a better fit to the data to predict log CST in preference to Model II. All of the values of TSS, temperature, paper type, and funnel type that were used to estimate the CST values experimentally were interpolated into Model I, and the 95% confidence intervals of the predicted estimates of CST were computed. The 479 estimates of CST used to calibrate the model were compared individually against the 95% prediction intervals (Table 1 Appendix). 461 out of the 479 observed estimates of CST were located within the lower and upper limits of the 95% prediction intervals, providing evidence for the validity of the model. 18 of the estimates were classified as outliers, located outside the 95% prediction intervals.

Figure 5.3.9 Distribution of residuals with respect to predicted values for Model I

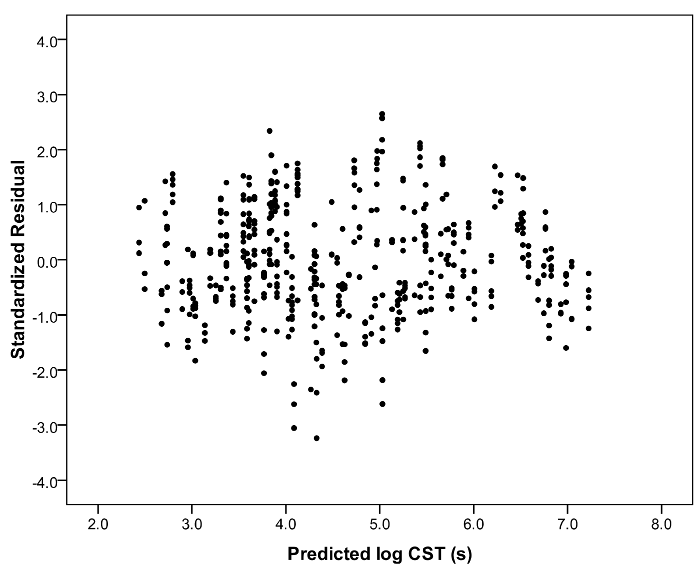


Figure 5.3.10 Normal distribution of residuals for Model I

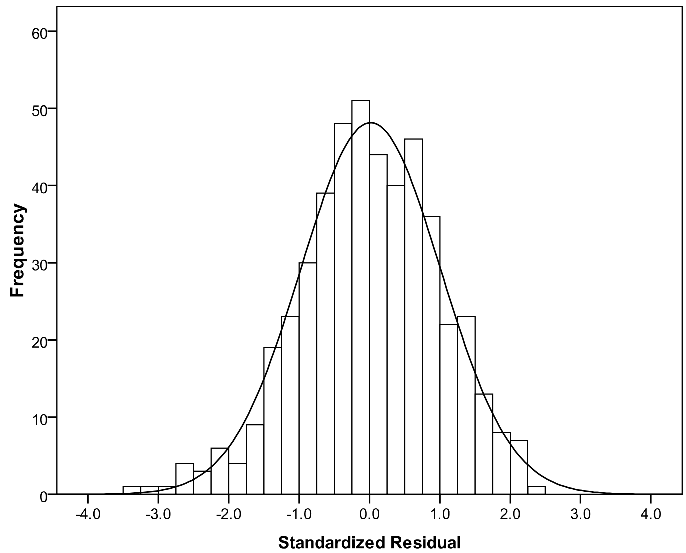
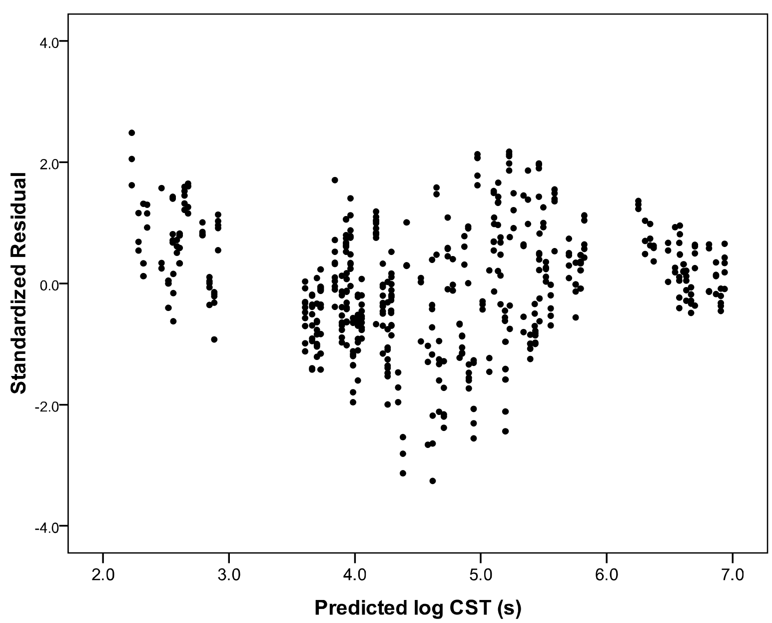


Figure 5.3.11 Distribution of residuals with respect to predicted values for Model II



The MLR was repeated excluding the 18 outliers in Model I to construct Model III (Table 5.3.6). Model III excluding outliers was defined by equation 5.3.3.

$$\log CST = 3.645 + 0.303\ TSS - 0.005\ TSS^2 - 0.197\ \text{temperature} + 0.006\ \text{temperature}^2 + 0.274\ \text{paper} - 0.226\ \text{funnel} \quad (5.3.3)$$

The accuracy and precision of Model III was an improvement on Model I, reflected by an increase in the R^2 value to 96.5%, and a reduction in the standard error to 0.232 (Table 5.3.6).

Table 5.3.6 MLR Model III to predict log CST, constructed by removal of the outliers from Model I
(a) ANOVA and R^2

Source of variance	Sum of Squares	Degrees of Freedom	Mean Square	F statistic	P	Adjusted R^2 (%)	Standard error of estimate
Regression	687.788	6	114.631	2130.2	0.000*	96.5%	0.232
Residual	24.592	457	0.054				
Total		463					

(b) Regression coefficients

Variable	Regression Coefficients	t statistic	P	95% confidence intervals of regression coefficients	
Intercept	3.645	28.498	0.000*	3.394	3.897
TSS	0.303	59.884	0.000*	0.293	0.313
TSS^2	-0.005	-36.171	0.000*	-0.005	-0.005
Temperature	-0.197	-12.943	0.000*	-0.227	-0.167
Temperature ^{2}	0.006	13.807	0.000*	0.005	0.007
Paper	0.274	12.512	0.000*	0.231	0.317
Funnel	-0.226	-10.493	0.000*	-0.268	-0.184

* Significant at $\alpha = 0.01$

5.3.2 Temperature and sludge formulation

The results of CST tests (mean CST (s) ± 95% confidence intervals) with respect to temperature (^{o}C) using 8 different formulations of synthetic sludges are presented in Figure 5.3.12. Lower CST estimates were obtained for sludges which did not contain KCl (formula 1, 2, 3, and 4) compared with sludges which did contain KCl (formula 5, 6, 7, and 8). The lowest CST estimates were for formula 1

(Alginate + $CaCl_2$) followed progressively by formula 2 (Alginate + $CaCl_2$ + Bentonite), formula 3 (Alginate + $CaCl_2$ + Bentonite + Kaolin) and formula 4 (Alginate + $CaCl_2$ + Kaolin). The CST estimates then increased progressively for formula 5 (Alginate + $CaCl_2$ + KCl); formula 6 (Alginate + $CaCl_2$ + Bentonite + KCl); and formula 7 (Alginate + $CaCl_2$ + Kaolin + KCl). The highest CST estimates were obtained using formula 8 (Alginate + $CaCl_2$ + Kaolin + Bentonite + KCl).

Multi-factorial ANOVA was applied to determine the effects of the sludge formula and the temperature on the mean CST estimates. Logarithmic transformations of the CST values were necessary to homogenize the variances and normalize the residuals. Levene's test indicated that the variances were homogeneous, and the Anderson-Darling test revealed that the residuals did not deviate from normality at the 0.01 level. The results of ANOVA indicated that the log CST estimates varied significantly at $\alpha = 0.01$ with respect to the different sludge formula at temperatures of between $10^{o}C$ and $25^{o}C$ (Table 5.3.7).

Figure 5.3.12 Results of CST tests (mean CST (s) ± 95% confidence intervals) with respect to temperature (^oC) using 8 different formulations of synthetic sludges (a) contains the following synthetic sludge formulas: 1 = Alginate + $CaCl_2$; 2 = Alginate + $CaCl_2$ + Bentonite; 3 = Alginate + $CaCl_2$ + Bentonite + Kaolin; 4 = Alginate + $CaCl_2$ + Kaolin and (b) contains the following synthetic sludges formulas: 5 = Alginate + $CaCl_2$ + KCl; 6 = Alginate + $CaCl_2$ + Bentonite + KCl; 7 = Alginate + $CaCl_2$ + Kaolin + KCl; 8 = Alginate + $CaCl_2$ + Kaolin + Bentonite + KCl

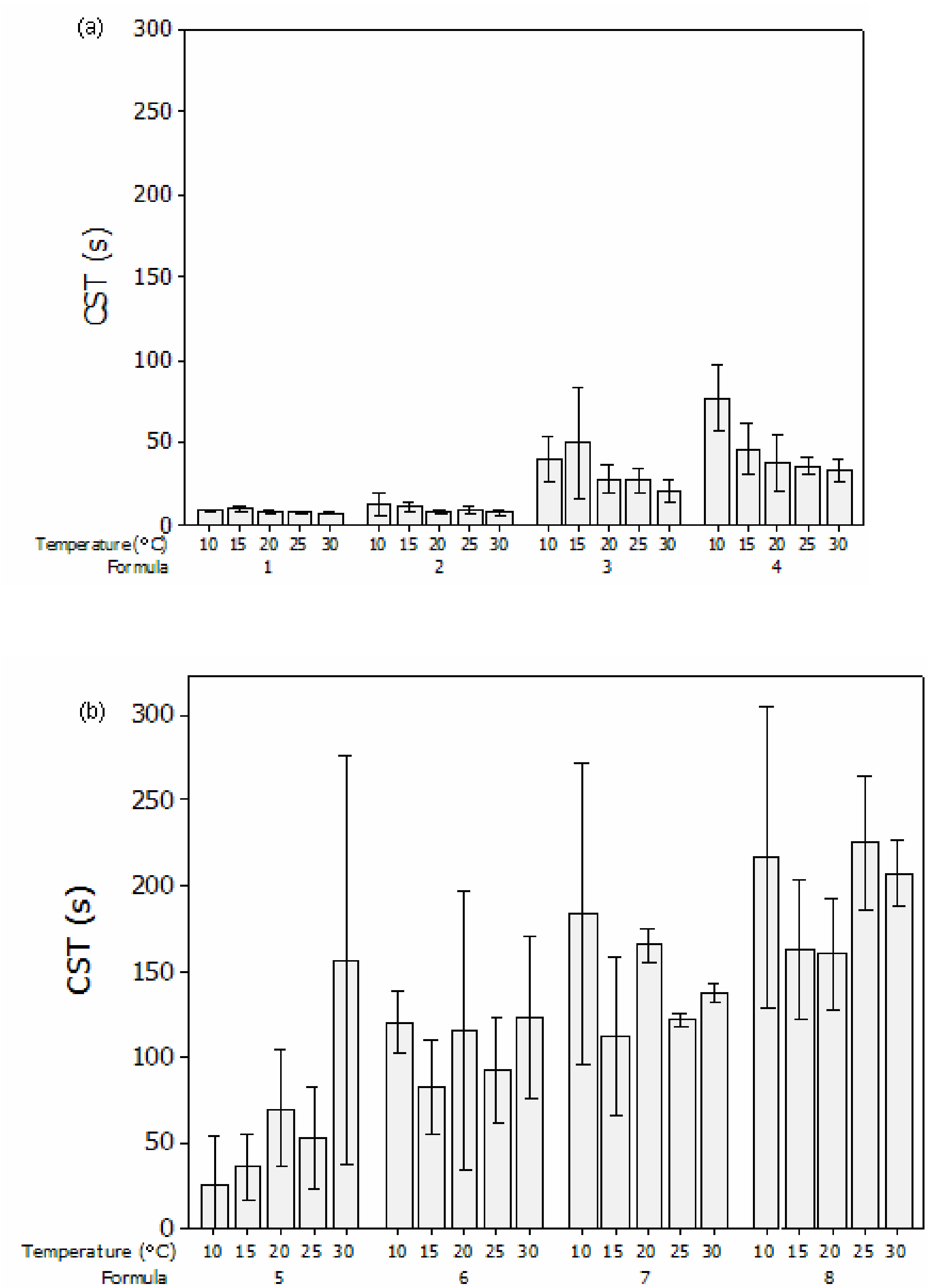
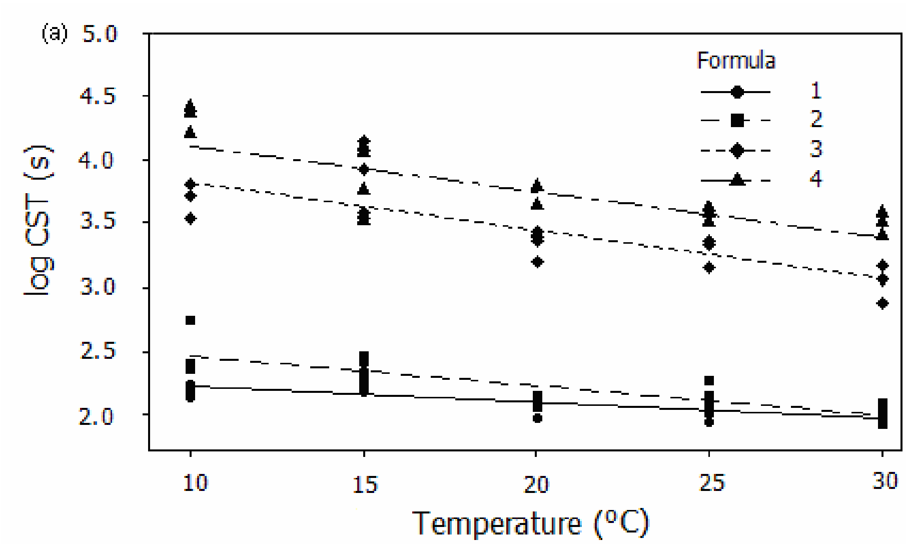


Figure 5.3.13 Interactions between log CST (s) and temperature using 8 different formulations of synthetic sludges (a) contains the following synthetic sludges formulas: 1 = Alginate + $CaCl_2$; 2 = Alginate + $CaCl_2$ + Bentonite; 3 = Alginate + $CaCl_2$ + Bentonite + Kaolin; 4 = Alginate + $CaCl_2$ + Kaolin (b) contains the following synthetic sludges formulas: 5 = Alginate + $CaCl_2$ + KCl; 6 = Alginate + $CaCl_2$ + Bentonite + KCl; 7 = Alginate + $CaCl_2$ + Kaolin + KCl; 8 = Alginate + $CaCl_2$ + Kaolin + Bentonite + KCl



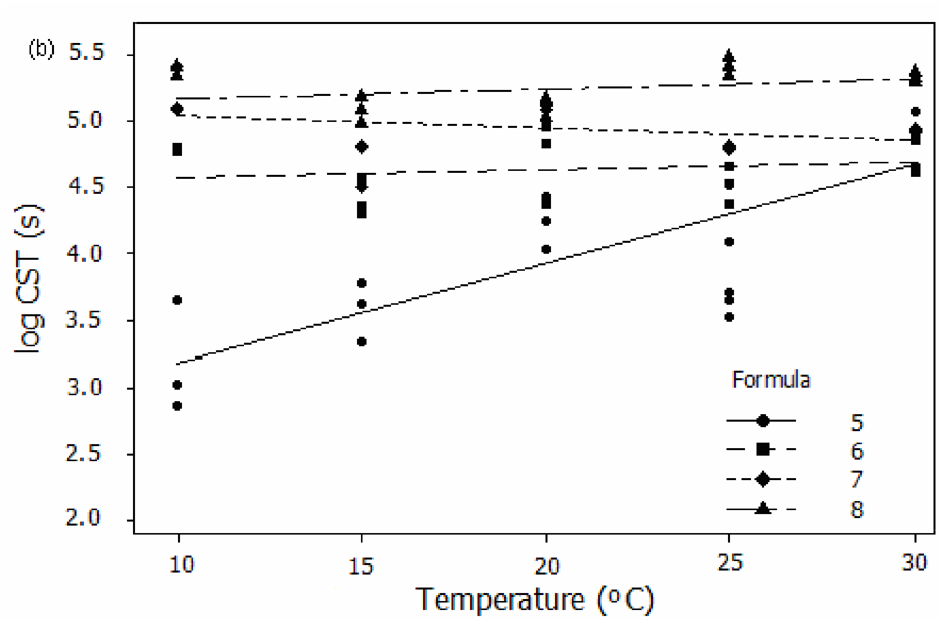


Table 5.3.7 Two-way ANOVA to compare the mean CST estimates with respect to sludge formula and temperature

Source	Sum of Squares	Degrees of Freedom	Mean Square	F	P
Temperature	2.142	4	0.535	11.38	0.000*
Formula	13.584	3	93.22	96.22	0.000*
Temperature x Formula	4.790	12	8.48	8.48	0.000*
Error	1.882	40			

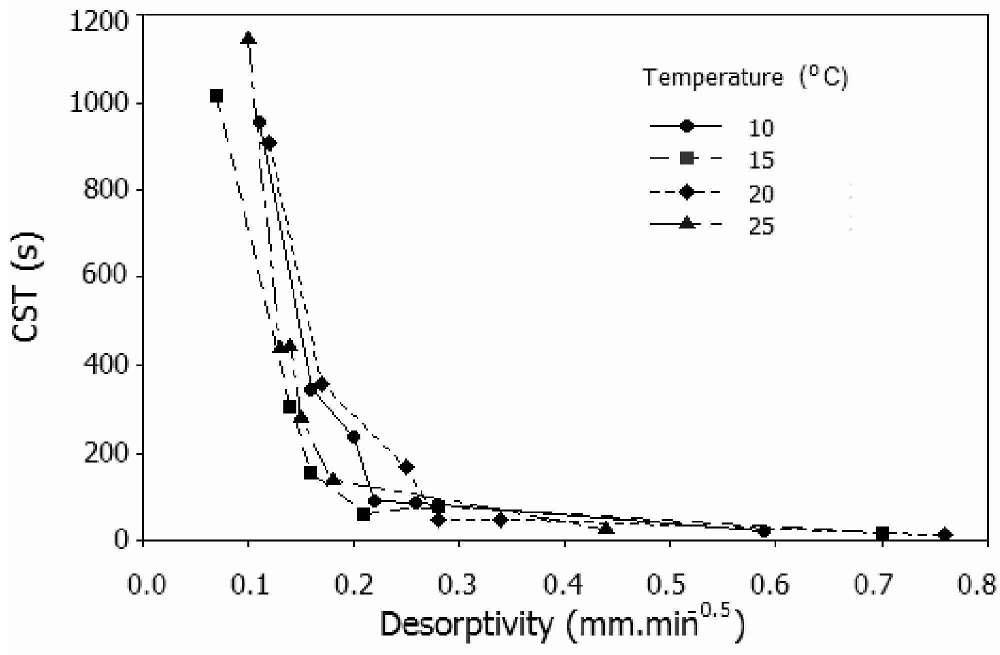
* Significant at $\alpha = 0.01$

A significant interaction between temperature and the sludge formula was revealed (Table 5.3.7). This interaction arose because the relationships between the CST estimates and the four temperatures varied depending on the compositions of the sludges. The logarithms of CST declined linearly with respect to the temperatures using the four sludges (formula 1, 2, 3, and 4) which did not contain KCl (Figure 5.3.13). Using sludge formula 5 (Alginate + $CaCl_2$ + KCl) however, the log CST increased linearly with respect to temperature. In contrast, the CST estimates did vary significantly with temperature using sludge formulae 7, and 8, which also included Kaolin and/or Bentonite (Figure 5.3.13). The CST estimates did not vary significantly for formulae 6.

5.3.3 Temperature and desorptivity

The synthetic sludge desorptivity estimated at temperatures of 10^oC to 25^oC and TSS concentrations of 2.3 to 31.6 g/l (Figure 5.3.14) was plotted against the mean CST estimates obtained at the same temperatures and sludge concentrations using standard CST apparatus (Whatman 17 chr paper and circular funnel).

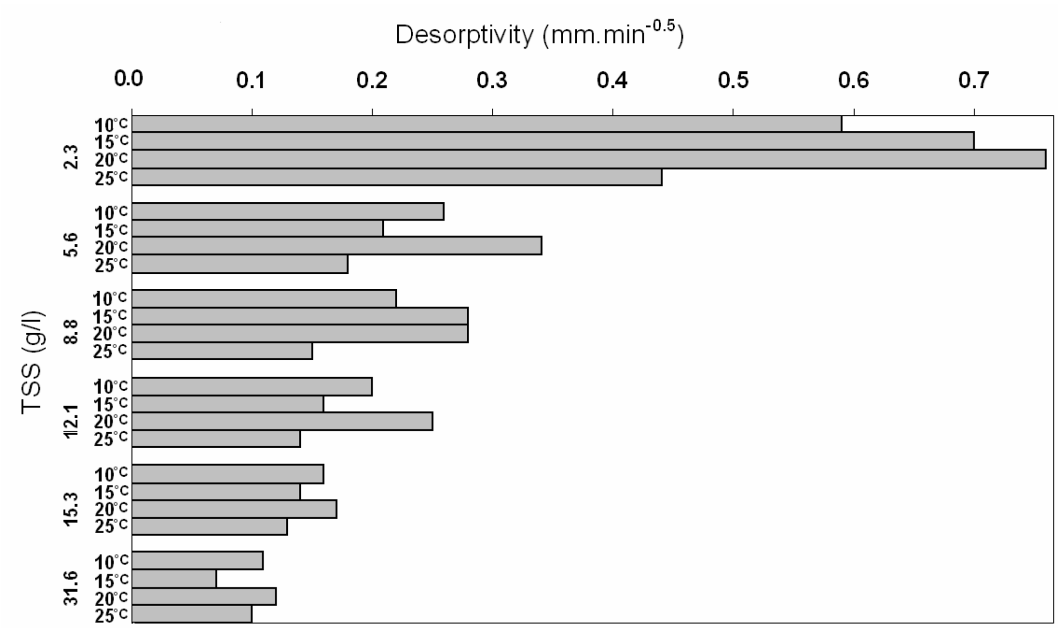
Figure 5.3.14 Relationships between the results of standard CST tests, the temperature and the desorptivity of synthetic sludge



The CST estimates at each temperature declined rapidly when the desorptivity increased from 0.1 and 0.3 mm.min$^{-0.5}$ but remained relatively constant when the desorptivity increased from 0.3 to 0.8 mm.min$^{-0.5}$ (Figure 5.3.14). The results of the CST tests were highly variable at high TSS concentrations when the water retention ability of the sludge was high (i.e., when the desorptivity was less than 0.3 mm.min$^{-0.5}$) but the CST estimates were relatively constant at lower TSS concentrations when the water retention ability of the sludge was lower, i.e. when the desorptivity exceeded 0.3 mm.min$^{-0.5}$.

The highest estimates of desorptivity were for the synthetic sample representing activated sludge (2.3 g/l TSS) and the lowest estimates were for the synthetic sample representing digested sludge (31.6 g/l TSS). The desorptivity of the synthetic sludges varied non-linearly with respect to the TSS concentrations and the temperature. Desorptivity displayed a trend to increase between temperatures of 10°C to 20°C, then to decrease between temperatures of 20°C to 25°C at each TSS concentration (Figure 5.3.15).

Figure 5.3.15 The relationships between desorptivity, TSS, and temperature



Linear relationships were visualized between the log desorptivity estimates and the log sludge concentrations at the four temperatures (Figure 5.3.16). Linearity implied that the logarithms of the sludge concentrations could be incorporated as a covariate in an ANCOVA model to determine the effect of temperature on desorptivity. Levene's test indicated that the variances were homogeneous, and the Anderson-Darling test revealed that the residuals did not deviate from normality at the 0.01 level. The results of ANCOVA indicated that the log desorptivity estimates varied significantly at $\alpha = 0.01$ between temperatures of 10°C to 25°C (Table 5.3.8).

These results confirmed the existence of significant relationships between sludge desorptivity, TSS, and temperature, which should be taken into account when interpreting the results of CST tests.

Figure 5.3.16 The relationships between log desorptivity, log TSS, and temperature

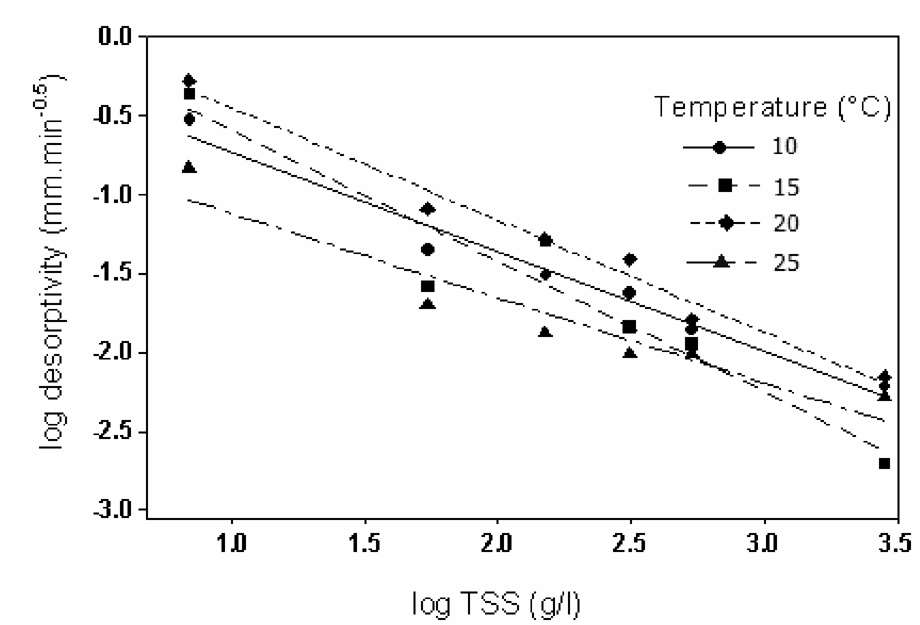


Table 5.3.8 Results of multi-factorial ANCOVA (with log TSS as a covariate) to determine the effects of variations in temperature on log desorptivity

Source	Sum of Squares	Degrees of Freedom	Mean Square	F statistic	P
log TSS (covariate)	7.3279	1	7.3279	218.68	0.000*
Temperature	0.6436	3	0.2145	6.40	0.004*
Error	0.6367	19	0.0335		
Total					

*Significant at $\alpha = 0.01$

The desorptivity of the synthetic sludges was found to vary non-linearly with respect to the TSS concentrations and the temperature. Synthetic activated sludge (2.3 g/l TSS) exhibited the highest desorptivity and hence the lowest water retention ability. Synthetic waste activated (8.8 g/l TSS) and digested sludge (31.6 g/l TSS) exhibited higher water retention ability. Consequently, it is expected that the results of CST test should decrease when the desorptivity of the sludge increases, whilst the CST should increase when desorptivity decreases.

5.4 Discussion

The results of CST tests at a constant temperature of 15^{o}C increased non-linearly with respect to the synthetic sludge concentrations between 2.3 g/l and 31.6 g/l using Fisher 200 chr papers and Whatman 17 chr papers with circular and rectangular funnels. These results implied that the relationships between the CST and the TSS had to be taken into account when modelling the relationship between CST and temperature. The relationship between the logarithms of the CST test results and the logarithms of the sludge TSS was found to be linear, therefore it was justified to incorporate log TSS as a covariate in ANCOVA.

When the temperatures of the sludge samples varied between 10^{o}C and 25^{o}C, the relationships between the mean CST values and the temperatures at each sludge concentration were non-linear. The mean CST values generally declined between 10-20^{o}C, then mostly increased between 20-25^{o}C. The coefficients of variation of the CST estimates did not display any clear trends with respect to the variations in temperature. The results of ANCOVA incorporating log TSS as a covariate revealed that temperature had a significant effect on the mean CST estimates. Multiple regression models were constructed which enabled the results of CST tests to be predicted using the TSS concentration, the temperature, the paper type, and the funnel type as predictor variables. The models required the use of logarithmic transformations and polynomial terms in order to simulate the observed non-linear behaviour. The models were empirical, and did not explain the physical and chemical processes that were responsible for causing variations in the results of CST tests with respect to temperature. These processes were assumed to be physical and chemical changes in the properties of the sludge associated with changes in temperature. Previous studies have indicated that the main physical effects of increasing the temperature of sludges are to reduce viscosity, reduce flocculation, reduce settleability and increase the density of small suspended particles (Dignac *et al.,* 1988; Chundakkadu and Loosdrecht, 1999; El Shafei *et al.* 2005). This study also found that increased temperature was responsible for reduction in the desorptivity.

The relationship between the logarithms of sludge viscosity and the reciprocal of temperature may be linear, as defined by the Arrhenius model (El Shafei *et al.* 2005). It was hypothesized that a linear relationship between the logarithms of the CST estimates and 1/temperature might indicate that variations in CST were a simple function of sludge viscosity. However, no such simple linear relationships were observed.

The change in the sludge viscosity and thereby the CST values associated with temperature could be due to other factors such as the surface tension between the filter paper and the surface of the plastic plate that is holding the filter paper, which could vary at different test temperatures (Baskerville and Gale, 1968; Ives, 1978). These results might suggest that CST results change by changing viscosity. Sludge is usually mixed during waste water treatment and this can affect its viscosity and thereby its dewaterability. These findings agree with previous results (Abu-Orf and Dentel, 1999), which showed that dewaterability is not the same at different sludge rheological properties. Mixing the sludge can change its rheological properties, and therefore its floc strength, which is a major factor in determining sludge dewaterability (Abu-Orf and Dentel 1999).

This study found that the desorptivity of the synthetic sludge displayed a general trend to increase between temperatures of 10^{o}C to 20^{o}C, then to decrease between temperatures of 20^{o}C to 25^{o}C at each TSS concentration. The results of ANCOVA indicated that the logarithms of desorptivity varied significantly between temperatures of 10^{o}C to 25^{o}C when log TSS was included as a covariate. The results of the CST tests were variable at high TSS concentrations when the water retention ability of the sludge was higher (i.e., when the desorptivity was less than 0.3 mm.min$^{-0.5}$) but the CST estimates were relatively constant at lower TSS concentrations when the water retention ability of the sludge was lower (i.e. when the desorptivity exceeded 0.3 mm.min$^{-0.5}$. Evidence was provided to conclude that relatively small variations in desorptivity and temperature could significantly influence the variability in the results of CST tests, particularly in sludges with high water retention ability. The results of this study indicated that changes in desorptivity associated with temperature had a significant influence on the observed variability in the results of the CST tests. Significant interactions between the results of CST tests, the temperatures and the sludge compositions were observed. The interactions arose because the relationships between the CST estimates and the temperatures varied depending on the compositions of the sludges. Potassium chloride in the presence of Kaolin and Bentonite was the most important ingredient responsible for promoting consistently high levels of CST estimated at temperatures between 10^{o}C and 25^{o}C. An increase in temperature from 10^{o}C to 25^{o}C was associated with an increase in the

CST estimates in the synthetic sludge consisting of only of Sodium alginate, Calcium chloride, and Potassium chloride. These results confirmed that the sludge dewaterability was strongly influenced by the presence of cations, specifically Potassium and Calcium (Sanin and Vesilind, 1999). The presence of cations was associated with a shift in the particle size distribution towards a higher frequency of larger particles between 400-700 µm in diameter (Figure 3.3.2), confirming the results of similar studies on the influence of cations on the flocculation of particles in synthetic sludges (Nguyen *et al.,* 2006; 2007; 2008). Flocculation is a process that occurs when suspended charged particles become loosely attached together, and coagulate to form fragile solid structures known as flocs (Dignac *et al.,* 1988; Chundakkadu and Loosdrecht, 1999). The results of this study indicated that changes in the flocculation properties of sludges associated with temperature had a significant influence on the observed variability in the results of the CST tests.

It is concluded that the non-linear relationships between the results of CST tests and the temperatures observed in this study, which were modelled empirically using multiple linear regression equations, were the consequence of multivariate interactions between temperature-controlled factors. These included variations in sludge viscosity, settleability, desorptivity, and particle size distribution, associated with variations in the flocculation behaviour of Bentonite, Kaolin, and Sodium Alginate induced by the presence of cations.

Chapter Six

Modelling the relationship between capillary suction time test (CST) and specific resistance to filtration (SRF)♣

6.1 Estimating filterability using the CST test

6.1.1 Introduction

It has been documented that there is relationship between the SRF and the rate at which the water travels between the electrodes of the CST device for a wide range of different types of sludge (Scholz, 2005). However, this relationship has not yet been quantified or modelled using appropriate empirical results, which is necessary to provide a platform for the investigation of this relationship in this study. The aim of this investigation was to explore the relationships between the four estimates of CST and the five distances between the electrodes in the CST test device by using linear regression models. The slopes of these lines were taken as estimates of the filterability, assumed to be a function of the SRF (see section 2.2, 2.8 and 2.9 for further details).

♣ The content of this chapter has been published in the following journal paper:

Sawalha, O., and Scholz, M. 2010. Modeling the relationship between capillary suction time and specific resistance to filtration. *Journal of Environmental Engineering-ASCE*, 136, 983-991.

6.1.2 Experimental design

The linear regression models were calibrated using data obtained from CST tests performed experimentally at three temperatures (15°C, 20°C and 25°C) and four total suspended solid (TSS) concentrations (8.8, 12.1, 15.3 and 31.6 g/l). Consequently, there were twelve experimental treatments (three temperatures x four TSS concentrations). Five replicates for each treatment were used to take the variance of the estimates into account. Therefore, a total of 60 samples were analyzed. The presence or absence of intercepts and the need for transformations were evaluated using four models, called A, B, C, and D:

$$Y = \beta_0 + \beta_1 X \qquad \text{(Model A)}$$

$$Y = \beta_1 X \qquad \text{(Model B)}$$

$$\log_e Y = \beta_o + \beta_1 \sqrt{X} \qquad \text{(Model C)}$$

$$\log_e Y = \beta_1 \sqrt{X} \qquad \text{(Model D)}$$

where Y = the predicted mean CST (s); β_0 = the intercept (the predicted mean CST value when the distance[4] between the stopping and starting electrodes of the CST device is zero); β_1 = the slope of the regression line (assumed to be a function of SRF, and called filterability); X = the distance[4] (m) between the electrodes of the CST device.

Logarithmic ($\log_e$) and square root transformations in Models C and D were justified following the application of Box-Cox tests. Logarithmic transformations may over-compensate a right-skewed distribution and create a left skew. In such a case, a square root transformation, which has less impact on right-skew, is the optimum transformation, in preference to logarithms (Tabachnick and Fidell, 2007). Consequently, both logarithmic and square root transformations were used to construct Models C and D. The transformation parameter for the X variable (distance^4) was constantly $\lambda = 0.50$ for the 12 treatments, implying that a square root transformation was appropriate (Table 6.1.1). The rounded transformation parameter for the Y variable (CST), however, was predominantly $\lambda = 0.00$, implying that a logarithmic transformation was appropriate. Consequently, it was decided to perform a square root transformation of the X variable (distance^4) and logarithmic transformations of the Y variable (CST). Since four models were applied, linear regression analysis generated four slopes (filterability estimates) for each treatment, denoted A, B, C and D as follows: A = slope of CST on distance^4 assuming the intercept was not zero**;** B = slope of CST on distance^4 assuming the intercept was zero**;** C = slope of log CST on $\sqrt{\text{distance}^4}$ assuming the intercept was not zero**;** D = slope of log CST on$\sqrt{\text{distance}^4}$ assuming the intercept was zero.

Table 6.1.1 Box Cox tests to determine the optimum transformations for linear regression of CST on distance^4 between electrodes

Treatment	TSS (g/l)	T (^oC)	Y (s) λ	Y 95% confidence intervals Lower limit	Y 95% confidence intervals Upper limit	Y Rounded λ	X (m^4) λ	X 95% confidence intervals Lower limit	X 95% confidence intervals Upper Limit	X Rounded λ
1	8.8	15	0.12	-0.27	0.49	0.00	0.42	-0.05	0.88	0.50
2	8.8	20	0.36	-0.05	0.77	0.50	0.42	-0.05	0.88	0.50
3	8.8	25	0.31	0.02	0.64	0.50	0.42	-0.05	0.88	0.50
4	12.1	15	0.03	-0.29	0.37	0.00	0.42	-0.05	0.88	0.50
5	12.1	20	0.36	-0.09	0.79	0.50	0.42	-0.05	0.88	0.50
6	12.1	25	0.17	-0.28	0.63	0.00	0.42	-0.05	0.88	0.50
7	15.3	15	0.39	0.00	0.75	0.50	0.42	-0.05	0.88	0.50
8	15.3	20	0.35	-0.06	0.76	0.50	0.42	-0.05	0.88	0.50
9	15.3	25	0.13	-0.69	0.50	0.00	0.42	-0.05	0.88	0.50
10	31.6	15	0.01	-0.32	0.37	0.00	0.42	-0.05	0.88	0.50
11	31.6	20	0.21	-0.28	0.70	0.00	0.42	-0.05	0.88	0.50
12	31.6	25	0.23	-0.27	0.71	0.00	0.42	-0.05	0.88	0.50

TSS total suspended solids; T Temperature; Y predicted mean CST; X distance^4 between electrodes; λ Box-Cox transformation parameter.

6.1.3 Results

The 48 estimates of filterability derived from the slopes of CST on distance4 between the electrodes using four linear regression models (A, B, C and D) for each of the 12 treatments are presented in Table 6.1.2.

Table 6.1.2 Linear regression of CST versus $distance^4$ between electrodes

Y	X	Model	Regression coefficients		T	P	R^2 (%)	Residual normality p
			Linear regression for Treatment 1 (Temperature = 15°C; TSS = 8.8 g/l)					
CST	$distance^4$	A	Intercept	-35.047	-3.25	0.004*	97.4	0.236
			Slope	6.469	29.38	0.000*		
		B	Slope (no intercept)	5.884	39.37	0.000*		0.005*
log CST	$\sqrt{distance^4}$	C	Intercept	2.404	31.93	0.000*	98.3	0.052
			Slope	0.436	36.72	0.000*		
		D	Slope (no intercept)	0.787	26.57	0.000*		0.066
			Linear Regression for Treatment 2 (Temperature = 20°C; TSS = 8.8 g/l)					
CST	$distance^4$	A	Intercept	-32.07	-3.29	0.003*	97.6	0.031
			Slope	6.097	30.67	0.000*		
		B	Slope (no intercept)	5.562	41.06	0.000*		0.152
log CST	$\sqrt{distance^4}$	C	Intercept	2.228	19.07	0.000*	96.3	0.106
			Slope	0.452	24.53	0.000*		
		D	Slope (no intercept)	0.778	27.77	0.000*		0.019*
			Linear regression for Treatment 3 (Temperature = 25°C; TSS = 8.8 g/l)					
CST	$distance^4$	A	Intercept	-135.16	-4.94	0.000*	96.3	0.010*
			Slope	13.626	24.44	0.000*		
		B	Slope (no intercept)	11.639	25.26	0.000*		0.005*
log CST	$\sqrt{distance^4}$	C	Intercept	1.939	16.17	0.000*	97.6	0.814
			Slope	0.574	30.35	0.000*		
		D	Slope (no intercept)	0.858	34.78	0.000*		0.010*
			Linear regression for Treatment 4 (Temperature = 15°C; TSS 12.1 g/l)					
CST	$distance^4$	A	Intercept	-118.72	-3.20	0.004*	94.9	0.006*
			Slope	15.627	20.68	0.000*		
		B	Slope (no intercept)	13.645	26.72	0.000*		0.005*
log CST	$\sqrt{distance^4}$	C	Intercept	2.913	36.04	0.000*	98.4	0.129
			Slope	0.475	37.27	0.000*		
		D	Slope (no intercept)	0.900	25.15	0.000*		0.096
			Linear regression for Treatment 5 (Temperature = 20°C; TSS = 12.1 g/l)					
CST	$distance^4$	A	Intercept	6.289	1.06	0.299^{ns}	99.6	0.031
			Slope	9.338	77.33	0.000*		
		B	Slope (no intercept)	9.443	135.8	0.000*		0.008*
log CST	$\sqrt{distance^4}$	C	Intercept	3.400	37.29	0.000*	96.7	0.005*
			Slope	0.376	26.12	0.000*		
		D	Slope (no intercept)	0.872	20.88	0.000*		0.028

Table 6.1.2 continued

Y	X	Model	Regression coefficients		T	P	R^2 (%)	Residual normality p
Linear regression for Treatment 6 (Temperature = 25°C; TSS = 12.1 g/l)								
CST	$distance^4$	A	Intercept	67.01	-1.88	0.073^{ns}	98.5	0.144
			Slope	28.707	39.43	0.000*		
		B	Slope (no intercept)	27.588	62.78	0.000*		0.012
log CST	$\sqrt{distance^4}$	C	Intercept	4.246	48.32	0.000*	97.3	0.043
			Slope	0.402	28.98	0.000*		
		D	Slope (no intercept)	1.022	19.65	0.000*		0.057
Linear regression for Treatment 7 (Temperature = 15°C; TSS = 15.3 g/l)								
CST	$distance^4$	A	Intercept	-92.57	-3.39	0.003*	98.7	0.526
			Slope	23.339	41.94	0.000*		
		B	Slope (no intercept)	21.794	56.89	0.000*		0.080
log CST	$\sqrt{distance^4}$	C	Intercept	3.684	32.81	0.000*	96.5	0.330
			Slope	0.443	25.02	0.000*		
		D	Slope (no intercept)	0.981	21.62	0.000*		0.059
Linear regression for Treatment 8 (Temperature = 20ºC; TSS = 15.3 g/l)								
CST	$distance^4$	A	Intercept	0.32	0.02	0.985^{ns}	99.3	0.288
			Slope	19.910	56.99	0.000*		
		B	Slope (no intercept)	19.916	101.42	0.000*		0.271
log CST	$\sqrt{distance^4}$	C	Intercept	4.093	41.43	0.000*	96.3	0.097
			Slope	0.382	24.52	0.000*		
		D	Slope (no intercept)	0.980	19.51	0.000*		0.048
Linear regression for Treatment 9 (Temperature = 25°C; TSS = 15.3 g/l)								
CST	$distance^4$	A	Intercept	-71.40	-1.43	0.166^{ns}	97.2	0.543
			Slope	28.809	28.34	0.000*		
		B	Slope (no intercept)	27.617	46.31	0.000*		0.012
log CST	$\sqrt{distance^4}$	C	Intercept	4.211	41.84	0.000*	96.6	0.853
			Slope	0.406	25.57	0.000*		
		D	Slope (no intercept)	1.021	19.76	0.000*		0.100
Linear regression for Treatment 10 (Temperature = 15°C; TSS = 31.6 g/l)								
CST	$distance^4$	A	Intercept	-972.4	-2.99	0.007*	91.5	0.207
			Slope	104.136	15.70	0.000*		
		B	Slope (no intercept)	87.904	20.01	0.000*		0.005*
log CST	$\sqrt{distance^4}$	C	Intercept	4.832	90.30	0.000*	99.2	0.674
			Slope	.462	54.68	0.000*		
		D	Slope (no intercept)	1.167	19.79	0.000*		0.057
Linear regression for Treatment 11 (Temperature = 20°C; TSS = 31.6 g/l)								
CST	$distance^4$	A	Intercept	104.39	1.39	0.179^{ns}	97.5	0.021
			Slope	45.726	29.77	0.000*		
		B	Slope (no intercept)	47.468	52.82	0.000*		0.005*
log CST	$\sqrt{distance^4}$	C	Intercept	5.176	55.28	0.000*	96.2	0.073
			Slope	0.356	24.04	0.000*		
		D	Slope (no intercept)	1.111	17.54	0.000*		0.076
Linear regression for Treatment 12 (Temperature = 25°C; TSS = 31.6 g/l)								
CST	$distance^4$	A	Intercept	75.27	1.05	0.305^{ns}	98.6	0.493
			Slope	59.125	40.39	0.000*		
		B	Slope (no intercept)	60.381	71.68	0.000*		0.698
log CST	$\sqrt{distance^4}$	C	Intercept	5.408	65.42	0.000*	97.0	0.837
			Slope	0.355	27.22	0.000*		
		D	Slope (no intercept)	1.145	17.32	0.000*		0.077

* Significant at $\alpha = 0.01$

All four regression models provided very good linear fits to the data. The R^2 values were between 91.5% and 99.6%. The t tests indicated that all the slopes were significantly different from zero at $\alpha = 0.01$. The corresponding values of the intercepts, however, were variable. Where intercepts were fitted, they were significantly different from zero in 6 out of the 24 models using untransformed data. All of the intercepts were significantly different from zero in the 24 models based on transformed data.

The Anderson-Darling tests indicated that the models varied with respect to violating the assumption of residual normality. The residuals deviated significantly from normality at the 0.01 level in 10 of the 48 models but did not deviate significantly from normality in the other 38 models.

This analysis indicated that the regression statistics were very variable with respect to the values of the intercepts and slopes, and the extent to which the residuals deviated from normality. It was therefore difficult to determine which model provided the best fit to the empirical data. An objective scoring system was established to determine the best fitting model. Considering the results for the four models and the twelve treatments collectively, one point was scored for each model, which had normally distributed residuals. The regression equation with the greatest number of points (scoring eleven out of twelve) was Model C (Table 6.1.3), which included a logarithmic transformation of Y and a square root transformation of X. The R^2 values of Model C were also very high, indicating that 96.2% to 99.2% of the variation in Y was explained by the variation in X. This justified the use of the slope β_1 of Model C to provide the most accurate estimates of filterability. Since the slope was $\log_e$ CST (measured in seconds) divided by $\sqrt{\text{distance}^4}$ (measured in m) the units of filterability using Model C become $\log_e$ s/m^2.

Table 6.1.3 Scores to identify the linear regression models with the best lines of fit (Scores = no significant deviation from residual normality)

Model	Treatment	Residual normality	Model	Treatment	Residual normality
A	1	ns	C	1	ns
(Score = 10)	2	ns	(Score = 11)	2	ns
	3	*		3	ns
	4	*		4	ns
	5	ns		5	*
	6	ns		6	ns
	7	ns		7	ns
	8	ns		8	ns
	9	ns		9	ns
	10	ns		10	ns
	11	ns		11	ns
	12	ns		12	ns
B	1	*	D	1	ns
(Score = 6)	2	ns	(Score = 10)	2	*
	3	*		3	*
	4	*		4	ns
	5	*		5	ns
	6	ns		6	ns
	7	ns		7	ns
	8	ns		8	ns
	9	ns		9	ns
	10	*		10	ns
	11	*		11	ns
	12	ns		12	ns

* Significant deviation from residual normality at $\alpha = 0.01$
ns No significant deviation from residual normality

The estimates of filterability (slopes of the regression lines) with respect to the four experimental TSS concentrations and the three temperatures using the four linear regression Models A, B, C and D are compared visually in Figure 6.1.1 and 6.1.2. The estimated filterability using Models A, B, and D generally increased systematically with respect to elevated TSS concentrations and temperatures. However, Model C (with fitted intercepts and transformed variables) described a more complex type of behaviour, in which the filterability estimate varied non-linearly with respect to temperature and TSS.

The intercepts were assumed, theoretically, to estimate CST when the distance between the electrodes was zero. The intercepts fitted for the Model A regression (using untransformed data) were both positive and negative, and appeared to reflect random error (Figure 6.1.3). In contrast, the intercepts fitted for Model C (using transformed data) were all positive and varied systematically with respect to the temperatures and the TSS concentrations The general pattern in Model C was for the intercepts to increase with respect to increased TSS concentrations at all three temperatures (Figure 6.1.3).

Figure 6.1.1 Filterability (slopes of regression lines) predicted by Model A and Model B (using untransformed variables) with respect to temperature and TSS concentration

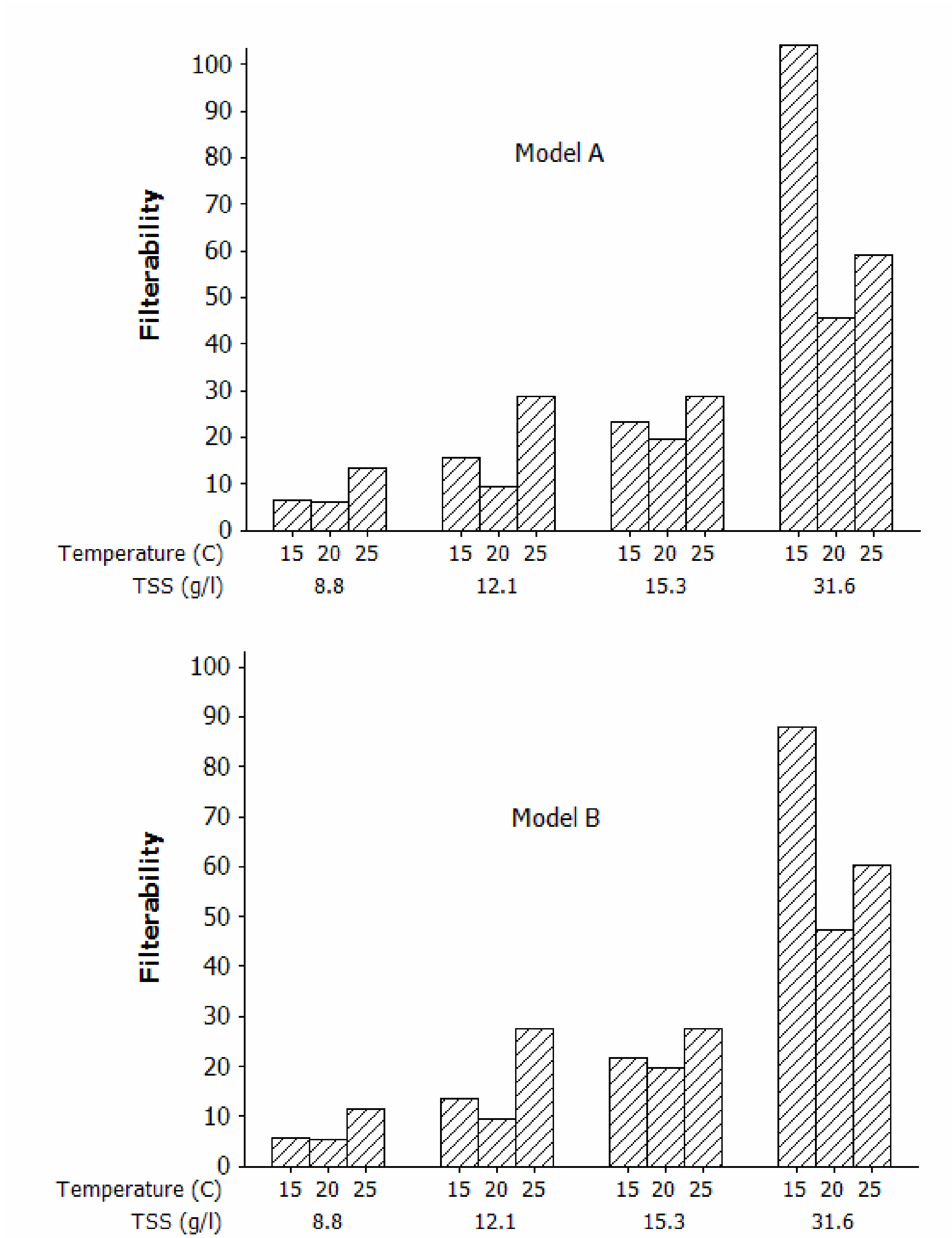


Figure 6.1.2 Log_e filterability (slopes of regression lines) predicted by Model C and Model D (using transformed variables) with respect to temperature and TSS concentration

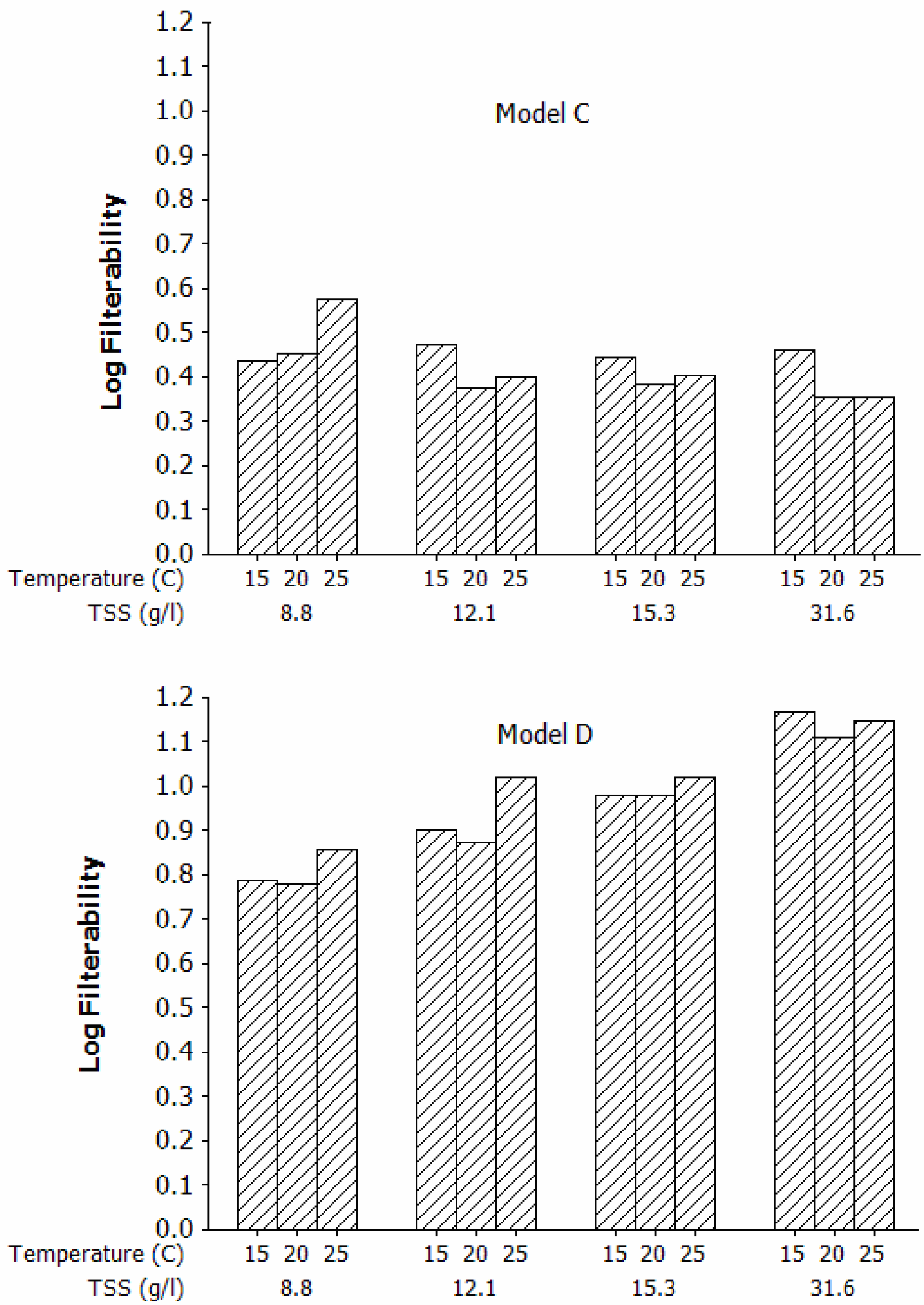
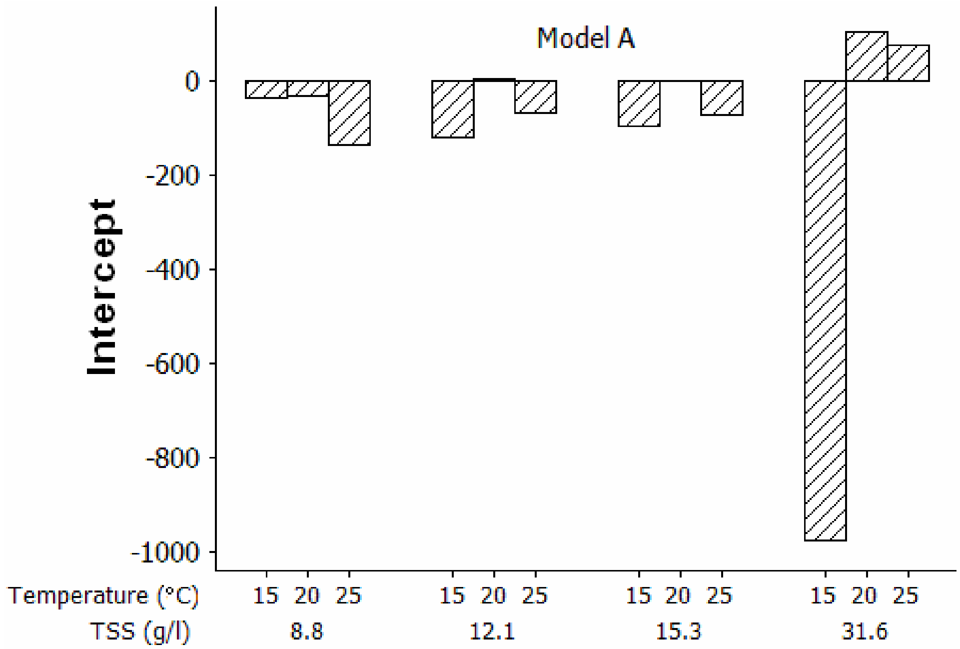
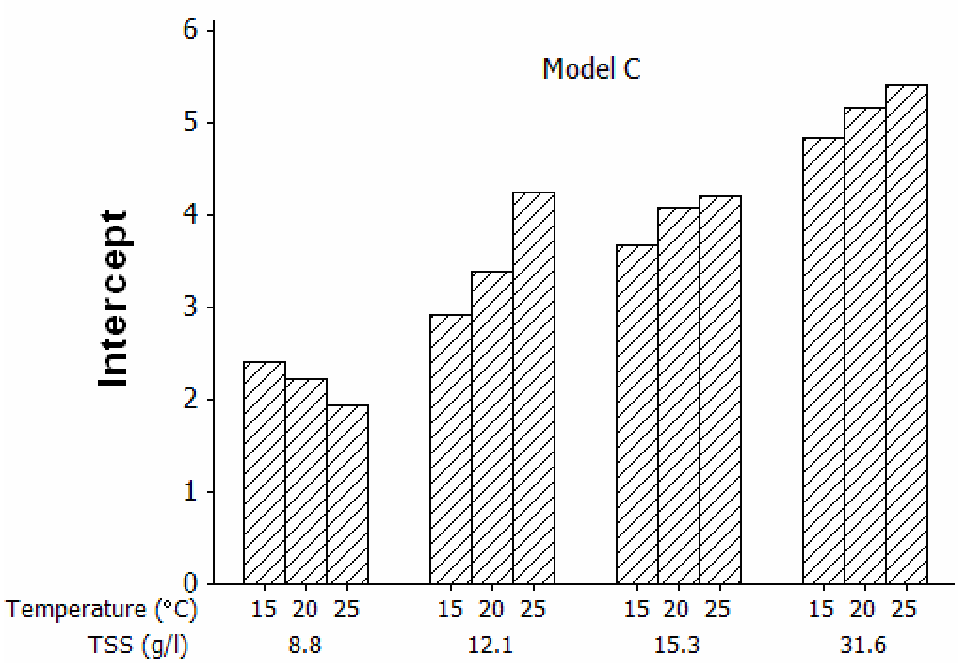


Figure 6.1.3 Intercepts of regression lines in Model A (using untransformed variables) and Model C (using transformed variables) with respect to temperature and TSS





6.1.4 Discussion

Four models were tested to estimate the filterability at four sludge concentrations and three temperatures, using the regression of the results of CST tests on the distance^4 between the electrodes. The regression models were very variable with respect to the values of the intercepts and slopes, and the extent to which the residuals deviated from normality. The model with the best line of fit, justified by its residual normality, high R^2 values, and consistent intercepts, was Model C, defined by equation 6.1.1.

$$\log Y = \beta_o + \beta_1 \sqrt{X} \qquad (6.1.1)$$

where Y = the predicted mean CST value (s); β_0 = the intercept (the predicted mean value of CST when the distance^4 between the stopping and starting electrodes of the CST device is zero); β_1 = the slope of the regression line (assumed to be a function of SRF, and called filterability); X = the distance^4 (m) between the electrodes of the CST device.

The practical application of this research is that it has modified the original assumption that filterability can be estimated as the simple linear regression of CST on the distance^4 between the electrodes (Meeten and Smeulders, 1995). The modifications were necessary, so that the real CST data could fit better the original assumption of Meeten and Smeldures (1995).

6.2 Modelling the relationship between CST and SRF

6.2.1 Introduction

The aim of this investigation was to construct a model to predict the results of specific resistance to filtration (SRF) tests using the results of (capillary suction time (CST) tests and two independent variables (temperature and sludge concentration).

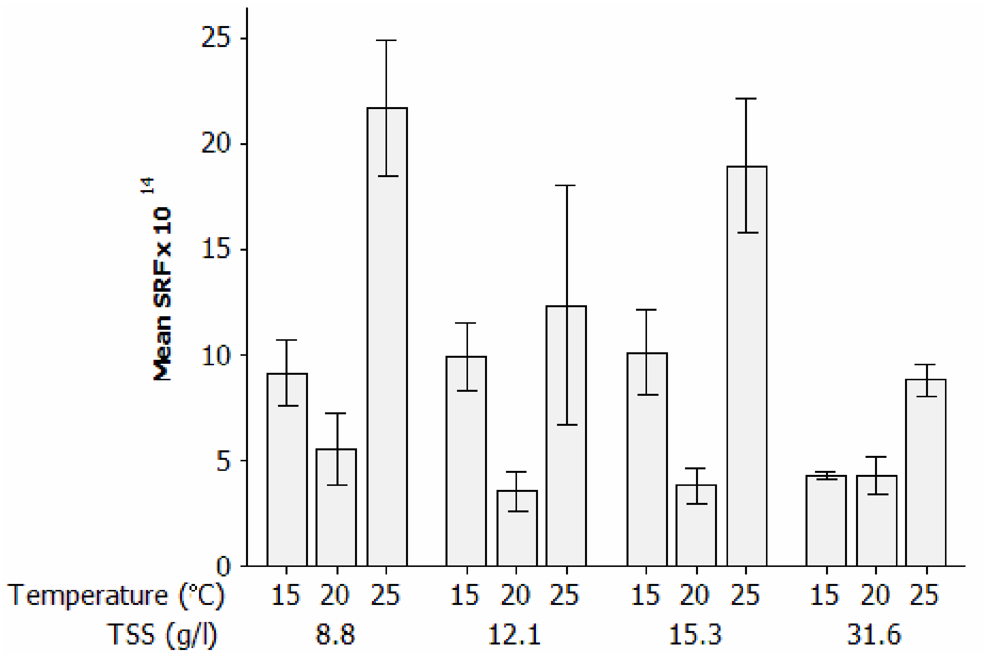
6.2.2 Experimental design

SRF tests were performed experimentally at three temperatures (15, 20, and 25^{o}C) four TSS concentrations of synthetic sludge (8.8, 12.1, 15.3 and 31.6 g/l). There were 5 to 16 replicates per SRF treatment. The mean SRF value, the coefficient of variation, and the 95% confidence intervals were computed for each treatment. MLR models were used to predict the mean SRF values for each treatment. The stepwise procedure was applied to identify an optimum sub-set of independent variables. Various transformations of filterability, TSS concentration, and temperature, were used. The model that best predicted the relationship between the results of the SRF tests and the CST tests was extracted from a total of 30 potential models.

6.2.3 Results

The experimental estimates of SRF were variable with respect to TSS and temperature (Figure 6.2.1). The general pattern was for the mean SRF to vary non-linearly with respect to TSS, and temperature. The highest mean SRF values were consistently at 25^{o}C, but there was a decrease in the mean SRF values between 15^{o}C and 20^{o}C. The widths of the 95% confidence intervals indicated that the SRF estimates varied within a group of replicates when the test was performed at the same TSS and temperature levels.

Figure 6.2.1 Mean values of SRF ± 95% confidence intervals estimated experimentally with respect to TSS and temperature



The non-linear behaviour between TSS, temperature (T), filterability (F), and the mean SRF values was simulated in MLR models by logarithmic and power transformations. Thirty multiple regression models, named C1 to C30, were constructed using the stepwise procedure to predict relationships between mean SRF, mean log SRF, T, T^2, TSS, TSS^2, log TSS, F, log F, and F/TSS (Table 6.2.1). The reason for the inclusion of F/TSS as an independent variable was to address the problem of co-linearity caused by the significant correlation ($r = -0.456$ at $\alpha = 0.01$) between filterability and TSS (Table 6.2.2).

Table 6.2.1 Models to predict SRF extracted by stepwise MLR using filterability (estimated with Model C), TSS and temperature as independent variables

Model	R^2 (%)	Mallow's Cp	SE	T	T^2	TSS	TSS^2	log TSS	F	log F	F/TSS
	Prediction of specific resistance to filtration (SRF)										
C1	24.4	21.0	5.15×10^{14}						X		
C2	23.9	21.1	5.16×10^{14}		X						
C3	64.1	6.4	3.55×10^{14}		X					X	
C4	62.6	6.9	3.62×10^{14}		X				X		
C5	76.4	3.2	2.88×10^{14}	X	X						X
C6	76.4	3.2	2.88×10^{14}	X	X					X	
C7	79.4	3.5	2.69×10^{14}	X	X		X		X		
C8	78.9	3.7	2.72×10^{14}	X	X		X			X	
C9	85.2	3.4	2.28×10^{14}	X	X	X	X				X
C10	84.2	3.6	2.35×10^{14}	X	X		X	X			X
C11	83.9	5.1	2.38×10^{14}	X	X	X	X				X
C12	83.6	5.1	2.40×10^{14}	X	X	X	X			X	X
C13	80.3	7.0	2.62×10^{14}	X	X		X	X	X	X	X
C14	80.3	7.0	2.63×10^{14}	X	X	X	X		X	X	X
C15	73.9	9.0	3.02×10^{14}	X	X	X	X	X	X	X	X
	Prediction of log specific resistance to filtration (log SRF)										
C16	18.7	56.8	0.55622						X		
C17	17.5	57.8	0.56031							X	
C18	67.0	17.7	0.35449	X	X						
C19	46.4	32.5	0.45182		X					X	
C20	81.8	7.6	0.26301	X	X						X
C21	81.5	7.8	0.26543	X	X	X					
C22	81.4	8.4	0.26642	X	X		X		X		
C23	81.2	8.5	0.26757	X	X		X				X
C24	83.2	8.0	0.25288	X	X	X	X				X
C25	82.5	8.4	0.25778	X	X		X	X			X
C26	86.0	7.6	0.23089	X	X	X	X		X		X
C27	85.7	7.7	0.23300	X	X		X	X	X		X
C28	89.5	7.4	0.20035	X	X	X		X	X	X	X
C29	88.3	7.7	0.21063	X	X		X	X	X		X
C30	87.5	9.0	0.21850	X	X	X	X	X	X	X	X

TSS Total suspended solids (g/l); T Temperature (ºC); F Filterability (log s/m^2)

Table 6.2.2 Correlations (Pearson's r correlation coefficients) between log SRF (m/g), filterability estimated with Model C, TSS and temperature.

Variable	Units	Log SRF	T	F
T	^{o}C	0.421*		
F	log s/m^2	0.511*	-0.136^{ns}	
F/TSS	log s/m^2/g/l	0.464*	0.024^{ns}	
TSS	g/l	-0.368^{ns}	0.000^{ns}	-0.456*

*Significant at α = 0.01
T Temperature; F Filterability; TSS Total Suspended Solids

Table 6.2.3 Optimum MLR model C20 to predict log SRF (m/g) using filterability (estimated with Model C), TSS, and temperature as independent variables

Variable	Units	Coefficient	Standard error	t statistic	P	Lower 95% confidence limit	Upper 95% confidence limit	F statistic	P
Intercept	m/g	46.128	2.535	18.194	0.000*	40.282	51.975	17.516	0.001*
T	^{o}C	-1.346	0.260	-5.175	0.001*	-1.945	-0.746		
T^2	^{o}C	0.035	0.006	5.416	0.001*	0.020	0.050		
F/TSS	log s/m^2/g/l	13.760	4.762	2.890	0.020*	2.779	24.742		

* Significant at α = 0.05
T Temperature; F Filterability; TSS Total Suspended Solids

Model C20 (Table 6.2.3) was selected as the best possible fit to the empirical data. The reasons for selecting this model, in preference to the other 29 models extracted by stepwise regression (Table 6.2.1), were that:

(a) All the partial regression coefficients of Model C20 were significantly different from zero at the 0.05 level; (b) Model C20 combined the least number of independent variables with a minimal value of Mallow's Cp (7.6), a low standard error (0.263), a high value of R^2 (81.8%) and a highly significant ANOVA (F = 17.516) at the 0.01 level; (c) Model C20 did not violate the assumptions of regression with respect to residual normality. The frequency of the residuals and the Anderson-Darling test p value = 0.371 indicated no significant deviation from residual normality (Figure 6.2.2); (d) The standardized residuals of Model C20 were randomly distributed around the mean (approximately zero) with no outliers or influence points. All standardized residuals were within two standard deviations of the mean (Figure 6.2.3).

Figure 6.2.2 Normal distribution of the standardized residuals for Model C20

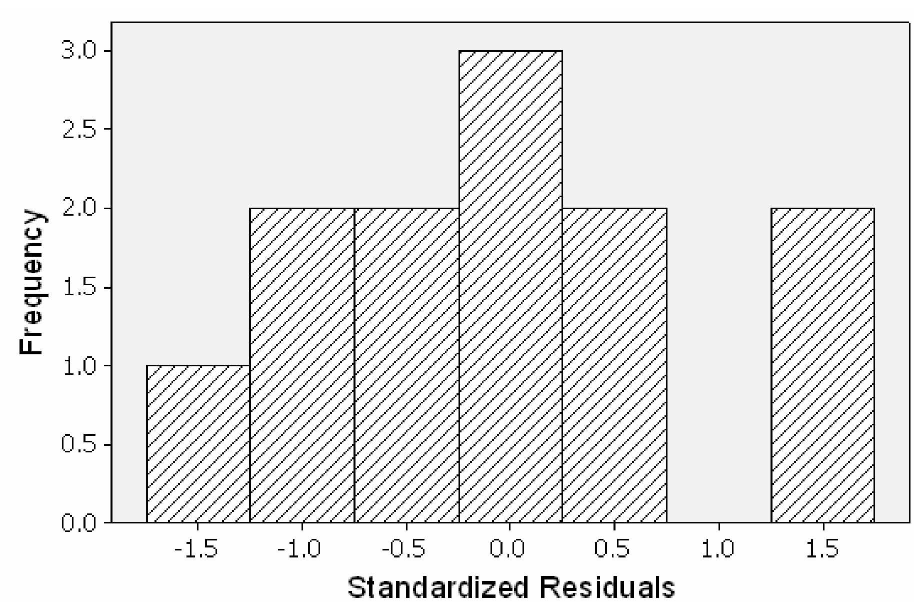
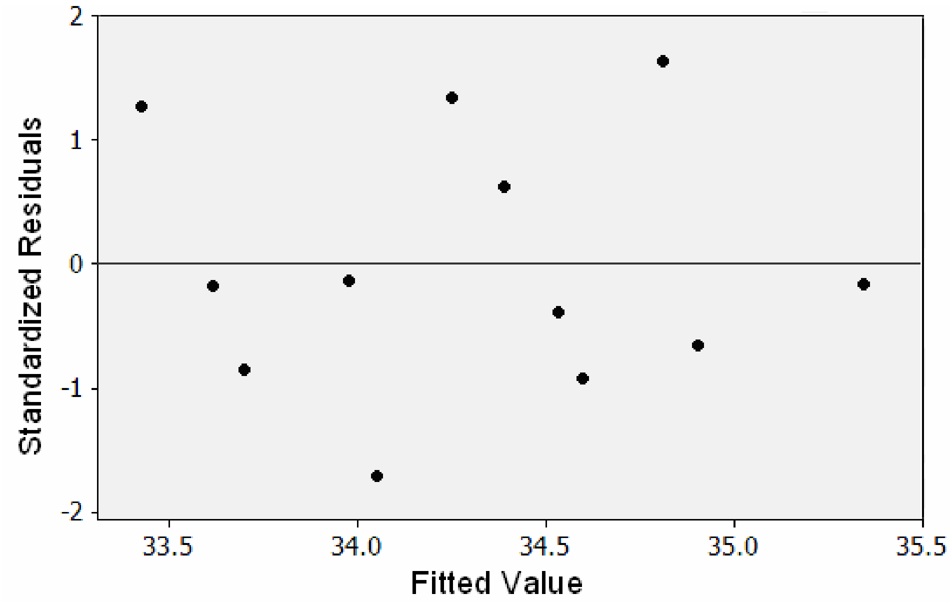


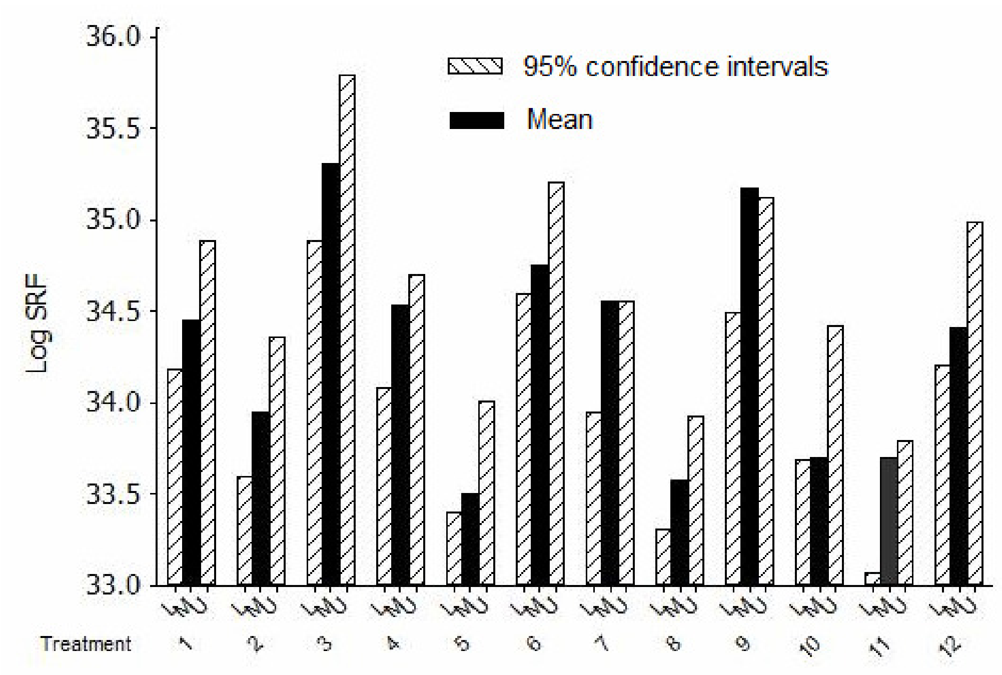
Figure 6.2.3 Distribution of the standardized residuals for Model C20 with respect to the fitted values



The co-linearity observed between filterability and TSS, indicated by a Pearson's correlation coefficient of -0.456 (Table 6.2.2) was eliminated by the incorporation of F/TSS as covariates in a combined independent variable.

All but one of the predicted mean log SRF values (for treatment 9) were within the upper and lower limits of the 95% confidence intervals for the mean log SRF values obtained experimentally (Figure 6.2.4).

Figure 6.2.4 Comparison of the predicted mean log SRF values with the 95% confidence intervals for the experimental mean log SRF values



L Lower limit of confidence interval; M Mean; U Upper limit of confidence interval

6.2.4 Discussion

The intention of the second part of this investigation was to construct multiple regression equations to predict SRF data using the best possible combination of independent variables, selected from filterability, temperature, and suspended solids concentration. This research did not, however, include filtrate viscosity as an independent variable, following the terms of equation 6.2.1 defined by Christensen et al. (1993). Neither was sludge viscosity included explicitly.

$$CST = c_1 SRF \mu_j W + c_2 \mu_j \qquad (6.2.1)$$

where c_1 and c_2 = coefficients related to the CST; μ_j = sludge viscosity; and W= solid content per unit volume of the filtrate.

The multiple regression models did incorporate sludge viscosity implicitly, since it is known that increased temperatures lead to a decrease in sludge viscosity, producing lower CST values (Ives, 1978). The relationship between sludge viscosity and temperature was investigated by El Shafei *et al.* (2005) who calibrated the Arrhenius equation empirically with respect to the viscosity and solid volume fraction of digested wastewater sludge (equation 6.2.2). The parameter K was defined empirically (equation 6.2.3).

$$\eta = K \times e^{1286/T} \qquad (6.2.2)$$

where η = sludge viscosity (Pa s); K = an empirical function of the solid volume fraction; and T = absolute temperature (ºC+273).

$$K = e^{107 \times C - 9.1} \qquad (6.2.3)$$

where K = an empirical function of the solid volume fraction; and C = solid fraction per unit volume of sludge.

Interpolation of equations 6.2.2 and 6.2.3 with the TSS concentrations and temperatures used in this investigation predicted an approximate doubling in the relative viscosity of sludge between TSS concentrations of 15.3 and 31.6 g/L, whilst an increase in temperature from 15^{o}C to 25^{o}C resulted in a relatively smaller decrease in viscosity. It is assumed, therefore, that variations in sludge viscosity influenced the variability in the CST and SRF estimates obtained in this investigation; however, the use of sludge and filtrate viscosity as explicit independent variables in multiple regression models to predict SRF data from the results of CST tests remains a need for future research.

In addition to viscosity, filterability depends on many physical properties of the CST test system. The CST varies with respect to TSS concentrations, not only because of variations in viscosity, but also because solids influence the thickness of the cake and its resistance to flow (Lee and Hsu, 1994b). Accordingly, Ives (1978) reported that at TSS concentrations of less than 5%, the magnitude of CST was similar to that of water alone, whereas at higher TSS concentrations, which produced thicker cakes (i.e. deeper depositions of solids onto the paper by sedimentation), the CST test provides a more accurate indication of filterability. Ives (1978) also included the filtration area, the net filtration pressure, and the specific resistance at the chosen net filtration pressure as factors which may control the magnitude of the CST.

Guan *et al.* (2003) and Scholz (2005) highlighted the importance of different floc sizes and structures to explain the variability in the dewaterability of sludge using CST tests. Cetin and Sürücü (1989) considered the influence of variable pH on the filterability and compressibility of sludge. The binding of variable amounts of water onto the filter is also important. It is apparent, therefore, that filterability and SRF are not only related to TSS, sludge viscosity, and temperature via the Arrhenius type equations (e.g. equations 6.2.2 and 6.2.3). The inter-relationships appear to be multi-variate, and non-deterministic. They may change from one test to another, depending on variable physical conditions, which may explain the residual variability in the approximations of filterability and the wide 95% confidence limits in the estimates of SRF observed in this study.

The mean values of the SRF test data varied in a complex non-linear way with respect to the four suspended solid concentrations (8.8, 12.1, 15.3 and 31.6 g/l) and the three temperatures (15, 20, and 25^{o}C) used in the 12 treatments. In view of the uncertainty surrounding the relationship between the results of the CST and SRF tests, it was necessary to explore numerous empirical models to select the best fit to the experimental data. The SRF data were best described using the following empirical model (equation 6.2.4), which incorporates logarithmic and squared terms to account for the non-linear behaviour, and includes the combined variable F/TSS to eliminate co-linearity between the filterability and the total suspended solids concentrations:

$$\log_e \text{SRF} = 46.128 - 1.346 \times T + 0.0351 \times T^2 + 13.76\ F/TSS \qquad (6.2.4)$$

where SRF = specific resistance to filtration (m/kg); T = temperature (ºC); F = filterability ($\log_e$ s/m^2/g/L) and TSS = total suspended solids (g/L).

All but one of the predicted mean $\log_e$ SRF values were within the upper and lower limits of the 95% confidence intervals for the mean $\log_e$ SRF values obtained experimentally. The practical application is that it has confirmed in mathematical terms the original assertion made by Baskerville and Gale (1968) that the results of CST and SRF tests are inter-related. It enables the SRF to be predicted from the results of CST tests, which may have practical value if SRF values are required but only the results of CST tests are available.

Chapter Seven

Practical implications of modified capillary suction time test (CST)

7.1 Introduction

The aim of this chapter was to explore the applications of the modified CST test based on the results of this study.

7.2 Practical implications

The main purpose of conducting CST and SRF tests at wastewater treatment plants is to save operating costs by evaluating the optimal dose of the sludge conditioner, defined as the dose of coagulant that yields the minimal capillary suction time or resistance to filtration (Wu *et al.*, 2000). Sludge conditioning results in the formation of dense flocs so that the solids can be more easily separated by subsequent dewatering processes. Conditioning is essential in order to improve the efficiency of the dewatering equipment, including rotary vacuum filters, centrifuges, drying beds, lagoons, filter presses, continuous belt filter presses, and thermal drying (Noyes, 1991). Sludge conditioning facilitates the separation of the solids from the liquid portion by dosing with inorganic salts (ferric chloride, lime, or Fenton-like reagents) or organic polymers (polyelectrolytes). Ferric chloride is the cheapest and is therefore most commonly used, and Aluminium sulphate is also widely used (Tony *et al*., 2008). Although organic polymers are more expensive, the amount of organic polymer required to produce the same volume of dried solids may be less, so the total cost of using organic polymers may work out about the same as using inorganic salts (Elliot, 2006). A high-energy rapid-mix to properly disperse the coagulant and promote particle collisions is needed to achieve the best results. Over-mixing does not affect coagulation, but insufficient mixing limits floc formation.

Selection of the correct coagulant dose requires laboratory testing under simulated plant conditions, followed by plant-scale trials. The dose-response relationship between the CST and the conditioner concentration is usually a U shaped curve. The minimal CST, at the lowest point of inflection of the curve, provides an estimate of the optimum conditioner dose. A reduction in the CST by over 50% can be achieved by optimal use of conditioners (Parker et al., 1972; Novak and Haugan, 1980; Guan et al., 2003; Hou and Li, 2003; Lai and Liu, 2004; Wang et al., 2005).

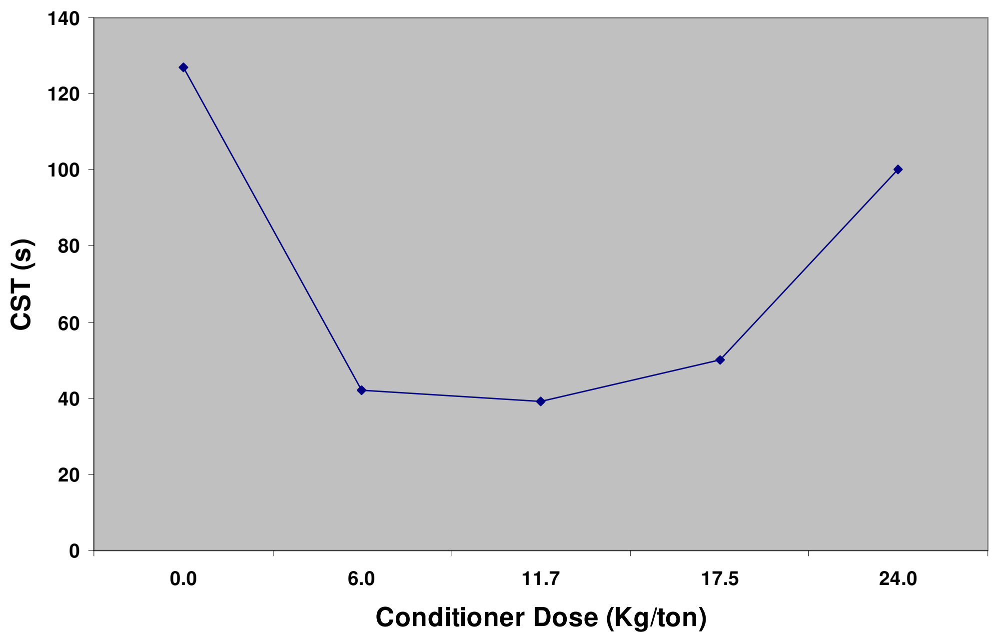
The total cost of sludge treatment at wastewater treatment plants is highly variable, mainly because of the different amounts of conditioner needed and the different methods used for dewatering (Ginestet, 2006). Lower operating costs are achieved if the most efficient conditioning and dewatering processes are used. Any evaluation of the conditioning process must take therefore into account the reduction in the cost of dewatering that can be achieved, offset against the cost of dosing with the conditioner (Ginestet, 2006). When applying different routes to optimize sludge treatment, it is possible to achieve considerable savings or losses simply through increased or decreased use of conditioners. In practice, it has been estimated that conditioners contribute an average of about 25-30% towards the cost of sludge dewatering; however, the cost of using conditioners can vary by as much as ± 10% depending on the amount and type of conditioner in use (Ginestet, 2006). Inefficient conditioner dosage is the major cause of excessive operating costs. If not made up to the correct dilution and dosed at the correct rate then the sludge may be wetter than optimum for the dewatering process and the plant operating costs are elevated. Inefficient conditioner dosage also implies wasted energy and more environmental damage. The cost of handling, hauling, and disposal of waste sludge from an industrial wastewater treatment plant is high and the cost of using polymer dosages exceed the 50% of overall sludge handling cost (Abu-Orf and Dentel, 1999).

A recent report (Watersense Inc., 2010) estimated current sludge disposal costs were "\$130.00 per cubic yard" (about £65 m^3). Because waste disposal requirements are becoming more restrictive, and because disposal sites are filling up, the cost of waste sludge disposal is likely to rise. It is therefore very important for wastewater treatment plants to minimize the amount of sludge that they generate. It may be possible for some plant managers to reduce costs simply by re-evaluating the use of coagulants, since the cost of sludge disposal can vary depending on the efficiency of the dewatering process (Watersense Inc., 2010). It is very important to ensure that the sludge conditioning and dewatering operations produces the driest possible sludge cake. The cost of handling and transportation is elevated if the sludge is not dried efficiently and its volume is greater than necessary. It is therefore crucial to keep the dosing system for the sludge conditioner at or near its optimum performance at all times (Naughton, 2004; Ginestet, 2006).

Accordingly, the results of this study have practical implications with respect to the interpretation of the dose-response curve of CST versus conditioner concentration. The interpretation of this curve controls the prescribed dose of conditioner, which in turn influences the plant operating costs. If the estimates of the CST are inaccurate and imprecise, then operating costs could be elevated. Figure 7.1.1 approximates the U shaped dose-response curve obtained by testing a waste activated sludge using a standard CST test device (Whatman 17 chr paper and circular funnel) against variable doses of a conditioner (electrolyte polymer NP-800+KP-201C) based on data in Lee and Liu (2000, p. 4432, Figure 2). It was not possible to extract the exact numbers from the published figure, and no standard deviations were provided to indicate the variability of the mean CST estimates. Consequently an approximated dose-response curve was constructed for the purposes of this discussion (Figure 7.2.1) based on the actual data provided by Lee and Liu (2000) as follows. The CST of the raw sludge was 127 seconds and the prescribed optimum conditioner dosage was 11.7 kg/ton dry solid when the minimal CST was 39 seconds. It was assumed for the purpose of this discussion that the optimum conditioner dose and the minimal CST were accurately and precisely measured by Lee and Liu (2000); however, this is unlikely to be true, given the variability of the CST test results demonstrated in this study and other studies. If the dose-response curve described by Lee and Liu (2000) was repeated in practice, it is unlikely that the estimated minimal CST would be 39 seconds, implying that the estimated prescribed optimum conditioner dose would also not be 11.7 kg/ton dry solids. The estimated CST would be greater or less than 39 seconds, depending upon many factors, including the experience of the person performing the test, the components of the

CST test device (e.g., the funnel geometry, the type of filter, whether or not a sealant was used), the test conditions, particularly the ambient temperature, as well as unknown random sources of variability.

Figure 7.2.1 Approximated dose-response curve of the CST versus conditioner dose



It is evident that in practice if the estimated minimal CST was greater than the expected minimal CST of 39 seconds (on the right hand side of the minimum point of the curve in Figure 7.2.1) then the prescribed conditioner dose would be excessively more than the optimum, leading to increased costs due to wasted conditioner. If the estimated CST was less than the expected minimal CST of 39 seconds on the left hand side of the curve then the prescribed conditioner dose would be insufficient, potentially leading to increased costs, since the subsequent sludge drying process could be ineffective.

The coefficients of variation of the CST estimates using a Whatman 17 chr paper and circular funnel for testing synthetic waste activated sludge in this study were about 20% (Table 5.3.1). Applying a 20% coefficient of variation to the minimal CST of 39 seconds gives a standard deviation of 7.8. A deviation in the estimated minimal CST from 39 to 47 seconds (about one standard deviation above the mean on either side of Figure 7.2.1). If overdosing this corresponds to a prescribed conditioner dose of about 16 kg/ton. This represents a conditioner overdose of over 4 kg/ton, representing approximately 33% more conditioner than necessary. It is estimated that (http://www4.ncsu.edu/unity/ lockers/users/k/kzering/ TFS2.html) that ferric chloride costs "$0.15 per pound" and polymer costs "$0.50 per pound" for use in water treatment processes (Assuming $1 = £0.64 and 1 pound = 0.454 kg, then ferric chloride costs £0.21 per kg and polymer costs £0.70 per kg). The mass of dry solids treated at large water treatment works can be up to about 19,600 tons/year (http://www.epcor.ca/en-ca/about-epcor/operations/operations-alberta/Edmonton /gold-bar/Pages/default.aspx). Assuming an optimum polymer conditioner dose of 11.7 kg/ton then the cost of conditioning 19,600 tons/year would be 19600 X 11.7 X 0.70 = £160,524; however, if an overdose of 16 kg/ton of polymer conditioner was applied, then the cost of conditioning would be 19600 X 16 X 0.70 = £219,520, an excess cost of £58,996. It is clear from these calculations that errors associated with estimating the CST to determine the optimum conditioner dose may considerably increase the cost of waste water treatment. This does not include the extra operation cost due to reduced dewaterability conditions associated with overdosing the conditioners.

The relationship between CST and temperature has an impact on evaluating conditioner dosage. The mean CST estimates in this study were found to generally decline between 10-20^{o}C but generally increased between 20-25^{o}C (Figure 5.3.2). The calibration and interpretation of dose-response curves to predict the optimum dose of conditioner could therefore be influenced by variations in temperature from one test to another, or from fluctuations in the ambient temperature in the laboratory when the CST tests were being performed. The CST of synthetic waste activated sludge was found to vary as much as 50% when the temperature varied by as little as 5^{o}C (Figure 5.3.2). This implies that if the dose-response curve in Figure 7.2.1 was calibrated using CST values estimated at 15^{o}C in the lab but the temperature of the sludge in the treatment plant was actually 20^{o}C then the CST of the plant sludge would be less, and the conditioner dose would be in excess. If the dose-response curve was calibrated when the lab sludge temperature was 20^{o}C but the temperature of the plant sludge was 25^{o}C then the minimal CST would be more than 39 seconds, and the conditioner dosage would be insufficient.

It is also important to take into consideration the components of the CST test device in order to calibrate the most precise dose-response curves. For example, it was shown that the CST estimates were consistently less variable when a sealant was used, depending on the type of filter and the funnel geometry (Table 4.4.1). A dose response curve calibrated using a CST device with a sealant is clearly not comparable to the curve obtained using a device without a sealant. When a sealant is used in the device, the estimate of CST is less variable, and so a more precise estimate of the optimum conditioner dose is possible, thereby saving operating costs at the treatment works.

Chapter Eight

Conclusions and Recommendations

8.1 Introduction

This chapter presents the conclusions and recommendations of the study with respect to the following nine objectives (Chapter 1):

1. To formulate a synthetic sludge to simulate the properties of natural sludge for experimental purposes;

2. To evaluate the applicability of altering the funnel geometries (e.g., circular or rectangular; smaller or larger;

3. To evaluate the applicability of several alternative filter papers (e.g., less expensive; isotropic; smaller pore diameter);

4. To evaluate the applicability of incorporating stirring activity in the test device;

5. To evaluate the applicability of adding a sealant at the bottom of the funnel, to eliminate or at least minimize unwanted filtrate leakage between the edge of the funnel and the filter paper, and potentially improve the test repeatability;

6. To determine the effects of temperature on the results of CST tests;

7. To model the effects of different sludge concentrations, temperatures, and methodological factors on the results of CST tests;

8. To model the relationships between the results of SRF tests and CST tests;

9. To recommend alternative methodologies that may help to improve the precision of the standard CST test device and procedures, and possibly reduce operational and consumable costs.

8.2 Synthetic sludges

Three synthetic sludges were formulated with similar properties to lighter natural sludges. 10% (2.3 g/l TSS) synthetic sludge approximated the properties of activated sludge; 30% (8.8 g/l TSS) synthetic sludge approximated the properties of waste activated sludge and 100% (31.6 g/l TSS) synthetic sludge approximated the properties of digested sludge.

TSS was found to be a major factor influencing the rheological properties of both synthetic and natural sludges, consistent with the conclusions of Dentel and Abu-Orf (1995) and Tixier *et al*. (2003). The sludge viscosities declined exponentially with respect to the TSS, and were typically a non-linear function of the shear rate. The effect of temperature on the viscosity varied with respect to the TSS. This study confirmed that due to their non-Newtonian characteristics, it was virtually impossible to predict the viscosity of sludge samples, given only the TSS and the temperature (Dentel, 1997; Forster, 2002; Tixier *et al*., 2003; Hasar *et al*., 2004). Other parameters such as the flocculation state, particle size, sludge composition, and bound water content, and density of filamentous bacteria were also considered to affect the sludge viscosity. Cellulose fibres were therefore included as essential components of the synthetic sludge to simulate the filamentous bacteria present in natural sludges, creating a large hysteresis area even at low TSS concentrations (Tixier *et al*., 2003).

The synthetic sludges contained different particle size distributions and flocculation properties. An elevated frequency of larger floc particles 400μm to 700 μm in diameter was associated with the addition of Calcium chloride and Potassium chloride. The mean particle size of 251 μm in the synthetic sludge was within the expected range for natural sludge (Jin *et al*., 2003); however, the floc sizes of synthetic sludges and natural sludges are not directly comparable, since the particle size distributions of natural sludges depend on the treatment plant operating conditions, including the flow rate, stirring rate, and flocculation behaviour. The influence of cations on the flocculation behaviour of Sodium alginate, Kaolin, and Bentonite particles in synthetic sludges (Nguyen *et al.* 2006; 2007; 2008) was confirmed in this study.

The overall conclusion of the studies on the properties of natural and synthetic sludges was that in order to interpret the results of CST tests a multitude of interacting sludge-specific factors, including the age, the temperature, the viscosity, the particle size distribution, the presence of cations, and the flocculation properties must be taken into account. Variations in these characteristics from one sample to another may be considered to be integral causes of the wide variability observed in the results of CST tests. The use of synthetic sludges was advantageous because they permitted experiments to be performed on stable samples with known physical and chemical properties not subject to degradation by microbial activity.

8.3 The effects of funnel geometry

It is concluded that the CST test times were consistently the highest and most variable using the 10 mm circular funnels, smaller and less variable when using the

18 mm circular funnels, and consistently the lowest and least variable when using the rectangular funnels. Irrespective of the type of filter paper used, the CST test results were consistently lower using rectangular funnels compared to circular funnels (except for three tests, when surplus sludge was tested using Carlson EE10H, MN280, and BF3 papers and FN30 when synthetic sludge was used.

The advantage of using a rectangular funnel was especially apparent when testing heavy gully pot and primary sludges. The highest and most variable CST estimates were obtained when testing heavy gully pot sludge using small 10 mm diameter circular funnels. The CST estimates for gully pot sludge declined and were less variable when 18 mm diameter circular funnels were used but declined even further and were the least variable when rectangular funnels were used.

It is concluded that the heavier gully pot and primary sludges were influenced more by sedimentation and cake resistance than the lighter activated sludges (Tiller and Li, 2001; Scholz, 2005). Another reason for reduced CST times could be that the rectangular funnels overcame the problem of anisotropic filter papers by making use of the linear flow in only one direction of the paper (Lee and Hsu, 1994b). When rectangular funnels were used there may have been a faster linear movement of the wet front through the filter papers, reducing the CST test times. In contrast, when circular funnels were used, the CST test times may have been higher because the wet fronts had an elliptical shape, so their movement was slower and non-linear.

8.4 The effects of filter papers

It is concluded that the filter paper type significantly influenced the results of the CST tests, after funnel size had been taken into account. The effects of the filter papers were particularly important when testing heavy sludges.

It has been suggested that the uneven movement of fluid across the diameter of anisotropic filters, compared with the even movement across isotropic papers, may influence the results of CST tests (Lee and Hsu, 1992). This study demonstrated, however, that there was no clear difference between the CST test results using isotropic and anisotropic papers. The isotropic papers HOVBO3 TO w/s, SS33205, and SS1107 generally produced lower CST estimates when testing gully pot and primary sludges, confirming that low CST test times are associated with some isotropic papers. Carlson EE10H, however, is also an isotropic paper, but it was consistently associated with high CST estimates. It is concluded, therefore, that the effects of filter papers on the results of CST tests were not only influenced by their isotropic or anisotropic properties. Other physical properties may influence CST test results including their porosity, weight, and thickness.

When testing heavy sludges, the CST estimates were negatively correlated with the pore sizes of the filters, and positively correlated with the basis weights and thicknesses of the filters. The highest CST estimates were obtained using a thick heavy filter with small pore diameter (i.e. Carlson EE10H) when testing primary and gully pot sludges. It is proposed that when testing heavy sludges using thick heavy filter papers with small pores, irrespective of whether or not the paper is isotropic or anisotropic, the CST may be elevated due to the high resistance to filtration associated with a rapid rate of blockage of the small pores by large particles, sedimentation, and dense cake formation. The CST may be lower when testing heavy sludges using thin light filter papers with a larger pore size (e.g. SS33205 and SS1107) due to the lower resistance to filtration associated with the lower rate of blockage of the pores, less sedimentation, and less cake formation. The correlation between filter paper pore size, thickness, and weight, and the results of CST tests was, however, not apparent when testing lighter surplus activated sludges, for which blockage, sedimentation, and cake formation are less of a problem than when testing heavy sludges. No correlation was found between the filter paper pore size, thickness, and weight, and the coefficients of variation of the CST test estimates. It might be inferred that the physical properties may not necessarily influence the variability in the results. However, this conclusion has to be taken with caution as the data used for this simple correlation analysis was very small and the due to the nature of the variables, the underlying assumptions might not be satisfied (most importantly normality and linearity).

The results of this study confirmed that Whatman 17 chr, which is commonly used filter paper for CST tests, did not produce the most consistent results in the shortest time. The CST estimates were frequently higher and more variable between replicates when using Whatman 17 chr papers than when using most other filter papers. The paper exhibiting the most desirable combination of low price, consistently low CST test times and low variability between replicates when testing heavy sludges, was SS1107, a thin light isotropic paper. The isotropic paper, H0VB0 T0 w/s was found to produce higher CST estimates when testing heavy gully pot sludge, but lower times with lighter sludges.

8.5 The effects of stirring

The suggestion that the results of CST tests may be improved by including a current induced by a stirrer within the sludge chamber to reduce or prevent sedimentation (Scholz, 2005) was not confirmed by this study. The results presented here are not consistent with this suggestion. Consequently the potential beneficial effects of stirring on the results of the CST tests could not be unequivocally proved.

It is possible that the current induced by the stirrer was insufficient to reduce or prevent sedimentation, particularly when testing the heavy gully pot and primary sludges, explaining why stirring had no statistically significant effects.

8.6 The effects of a sealant

The results of the CST tests with and without a sealant depended on the funnel geometry and the filter paper type. When the sealant was used, the highest mean CST values were obtained using Whatman 17 chr paper and a circular funnel, compared with the lower values obtained using Fisher 200 chr paper and a rectangular funnel. When the sealant was not used, the highest mean CST estimates were also observed using Whatman 17 chr paper and a circular funnel, compared with the lower values obtained using Fisher 200 chr paper and a rectangular funnel. Irrespective of the type of filter paper or funnel, the slopes of the linear regression lines of the CST on the sludge concentration (i.e., the rates at which the CST estimates increased per unit increase in sludge concentration) were consistently higher when the sealant was used, compared to when the sealant was not used.

The use the sealant reduced the variability (coefficients of variation) observed in the results of replicated CST tests. The coefficients of variation were higher when no sealant was used (3.4% to 32.5%) compared to when there was a sealant (2.5% to 19.1%). The coefficients of variation varied non-linearly with respect the sludge concentrations, and could not be predicted using regression models.

These results are not comparable with those of any other researchers, since no previous studies have been performed on the effects of using a sealant on the results of CST tests.

8.7 The effects of temperature

Baskerville and Gale (1968) warned that the results of CST test were sensitive to variation in temperature. They suggested that to correct the CST values for testing temperature by using a correction factor of the ratio of the water at the testing temperature to a standard temperature. Despite this warning, little research had been done to describe or predict the effects of the variation in temperature on the results of CST tests. This study concluded that temperature has a significant effect on the CST estimates, but the relationship is complex, precluding the computation of a simple correction factor. When the temperatures of the synthetic sludge samples varied between 10^{o}C and 25^{o}C, the relationships between the mean CST values and the temperatures at each sludge concentration were non-linear. The mean CST values generally declined between 10-20^{o}C, then mostly increased between 20-25^{o}C. The coefficients of variation did not display any clear trends with respect to the temperature.

Linear relationships between the logarithms of the CST test results using synthetic sludges and 1/temperature, which might indicate that the variations in CST were a function of sludge viscosity following the Arrhenius equation (El Shafei *et* *al.,* 2005) were not observed. The change in the sludge viscosity and thereby the CST values associated with temperature could be due to other factors such as the surface tension between the filter paper and the surface of the plastic plate that is holding the filter paper, which could vary at different test temperatures (Baskerville and Gale, 1968; Ives, 1978). These results might suggest that CST results change by changing viscosity. Sludge is usually mixed during waste water treatment and this can affect its viscosity and thereby its dewaterability. These findings agree with previous results (Abu-Orf and Dentel, 1999), which showed that dewaterability is not the same at different sludge rheological properties. Mixing the sludge can change its rheological properties, and therefore its floc strength, which is a major factor in determining sludge dewaterability (Abu-Orf and Dentel 1999).

8.8 The prediction of CST test results

Multiple regression models were constructed which predicted the CST test results using the TSS concentration, the temperature, the paper type, and the funnel type as predictor variables. The models required the use of logarithmic transformations and polynomial terms in to simulate non-linear behaviour.

The best fitting empirical model to predict the results of CST tests from temperature, paper type, and funnel geometry, excluding outliers, was:

$$\log CST = 3.645 + 0.303\ TSS - 0.005\ TSS^2 - 0.197\ temperature + 0.006\ temperature^2 + 0.274\ paper - 0.226\ funnel$$

where CST = capillary suction time (s); TSS = total suspended solids (g/l); temperature = ^{o}C; Fisher 200 chr paper = 0; Whatman 17 chr paper = 1; circular funnel = 0; rectangular funnel = 1.

The CST test times decreased when the desorptivity of the sludge increased, but increased when desorptivity decreased. The desorptivity of the synthetic sludge generally increased between temperatures of 10^{o}C to 20^{o}C, then decreased between temperatures of 20^{o}C to 25^{o}C at each TSS concentration. Evidence was provided to conclude that relatively small variations in desorptivity and temperature could significantly influence the variability in the results of CST tests, particularly in sludges with high water retention ability. Significant interactions between the results of CST tests, the temperatures and the sludge compositions were observed. The interactions arose because the relationships between the CST estimates and the temperatures varied depending on the compositions of the sludges. Potassium chloride in the presence of Kaolin and Bentonite was the most important ingredient responsible for promoting consistently high levels of CST estimated at temperatures between 10^{o}C and 25^{o}C. In contrast, an increase in temperature from 10^{o}C to 25^{o}C was associated with an increase in the CST estimates in the synthetic sludge consisting of only of Sodium alginate, Calcium chloride, and Potassium chloride. It was concluded that the sludge dewaterability was strongly influenced by the presence of cations, specifically Potassium and Calcium (Sanin and Vesilind, 1999) associated with a shift in the particle size distribution towards a higher frequency of larger particles between 400-700 µm in diameter. The results of this study indicated that changes in the flocculation properties of sludges associated with temperature had a significant influence on the observed variability in the results of the CST tests. These results were consistent with those of similar studies on the influence of cations on the flocculation of particles in synthetic sludges (Nguyen *et al.,* 2006; 2007; 2008).

It is concluded that the non-linear relationships between the results of CST tests and the temperatures observed in this study, were the consequence of multivariate interactions between temperature controlled factors. The models were empirical, and did not explain the physical and chemical processes that were responsible for causing variations in the results of CST tests with respect to temperature. These processes were assumed to be physical and chemical changes in the properties of the sludge associated with changes in temperature, including the reduced viscosity, the reduced flocculation, the reduced settleability, the reduced desorptivity, the elevated density of small suspended particles, and variations in the flocculation behaviour of Bentonite, Kaolin, and Sodium Alginate induced by the presence of cations (Dignac *et al.,* 1988; Chundakkadu and Loosdrecht, 1999; El Shafei *et al.* 2005).

8.9 The prediction of filterability

The original assumption that filterability can be estimated as the simple linear regression of CST on the distance^4 between the electrodes (Meeten and Smeulders, 1995) was modified by this study. A logarithmic transformation of the CST test times and a square root transformation of the distance^4 between the electrodes was found to improve the fit of the regression line to the data.

8.10 The prediction of SRF test results

It is concluded that SRF test results can be predicted from the results of CST tests using an empirical model in which the temperature, the filterability, and the TSS are predictor variables. The model developed based on an empirical data, and confirmed the assumption that the results of CST and SRF tests are inter-related (Baskerville and Gale, 1968).

8.11 Practical implications

The main purpose of conducting CST test at wastewater treatment plants is to reduce operating costs by evaluating the optimal dose of the sludge conditioner, defined as the dose of coagulant that yields the minimal capillary suction time or resistance to filtration (Wu *et al.*, 2000). Accordingly, the results of this study have practical implications with respect to the interpretation of the dose-response curve of CST versus conditioner concentration. The interpretation of this curve controls the prescribed dose of conditioner, which in turn influences the plant operating costs. If the estimates of the CST are inaccurate and imprecise, then operating costs could be elevated. More details about practical implication are given in Chapter 7.

8.12 Recommendations

The results of this study confirmed the recommendation (Scholz, 2005) that the larger 18 mm diameter circular funnels, and preferably the rectangular funnels, should be used to test heavy sludges because the larger funnels significantly reduce the variability and the time taken to conduct the CST tests.

It is recommended that the widespread use of anisotropic Whatman 17 chr papers for CST tests cannot be justified. Whatman 17 chr, which is the most commonly used filter paper for CST tests, did not produce the most consistent results in the shortest time. It is recommended that Isotropic filter papers should be used instead of Whatman 17 chr, in order to lower the cost, reduce the CST test times and improve the precision of the estimates. SS1107 is considered to be most suitable for testing heavy sludges.

It is recommended that a sealant should be used in the CST test apparatus to reduce the leaks between the funnel and the paper, and improve the precision of the results, by lowering the variability between replicate tests.

The users of CST tests must consider the complex non-linear relationships that exist between the CST test times and the temperature. The physical and chemical properties of sludges are strongly influenced by temperature, including significant effects on viscosity, settleability, desorptivity, and flocculation behaviour. The practical implications are that the CST test results obtained using one sample of sludge at one temperature are not necessarily comparable with the results obtained on the same sample of sludge at another temperature. It is recommended that the temperature should be recorded, and should be carefully controlled during the conduction of all CST tests.

The following model is recommended to predict filterability:

$$\log Y = \beta_o + \beta_1 \sqrt{X}$$

where Y = the predicted mean CST value (s); β_0 = the intercept (the predicted mean value of CST when the distance[4] between the stopping and starting electrodes of the

CST device is zero); β_1 = the slope of the regression line (assumed to be a function of SRF, and called filterability); X = the distance^4 (m) between the electrodes of the CST device. The practical application of this model is that it modifies the original method, to predict filterability as the simple linear regression of CST on the distance^4 between the electrodes (Meeten and Smeulders, 1995).

It is recommended that SRF test results can be predicted from the results of CST tests using the model:

$$\log \mathrm{SRF} = 46.128 - 1.346\ T + .035\ T^2 + 13.760\ F/TSS$$

where SRF is the specific resistance to filtration (m/kg); T is the temperature (ºC); F is the filterability ($\log s/m^2$) and TSS is the total suspended solids concentration (g/l). This model predicts the SRF from the results of CST tests, which may have practical value if SRF values are required but only the results of CST tests are available; however, the model predicts SRF for the suspended solid concentrations within the range of the solid content were used in this study.

To save money and energy, and to reduce environmental damage, it is crucial for wastewater treatment plant managers to ensure that the dosing system for the sludge conditioner is at or near its optimum performance at all times. It may be possible for some plant managers to reduce costs simply by re-evaluating the use of conditioners, based on their interpretation of the results of CST tests. It is recommended that users of dose-response curves which predict the optimum dose of conditioner are aware that the variability in the optimum conditioner dose using the CST test may be influenced by differences in the components of the CST test device from one test to another (e.g. funnel geometry, filter type, and use of a sealant) and by fluctuations in ambient temperature.

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Appendix

Table 1 Comparison of the observed CST estimates with the upper and lower 95% limits of the CST predicted by MLR Model I

Temperature (°C)	TSS (g/l)	Paper type	Funnel Geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
10	2.3	1	0	21.8	15.6	43.1	*
10	2.3	1	0	23.0	15.6	43.1	*
10	2.3	1	0	21.6	15.6	43.1	*
10	2.3	1	0	21.4	15.6	43.1	*
10	2.3	1	0	21.4	15.6	43.1	*
10	5.6	1	0	51.2	37.2	102.8	*
10	5.6	1	0	90.8	37.2	102.8	*
10	5.6	1	0	88.2	37.2	102.8	*
10	5.6	1	0	94.2	37.2	102.8	*
10	5.6	1	0	83.6	37.2	102.8	*
10	5.6	1	0	92.4	37.2	102.8	*
10	5.6	1	0	85.2	37.2	102.8	*
10	5.6	1	0	86.0	37.2	102.8	*
10	5.6	1	0	97.0	37.2	102.8	*
10	5.6	1	0	91.8	37.2	102.8	*
10	5.6	1	0	86.0	37.2	102.8	*
10	8.8	1	0	88.8	76.5	211.2	*
10	8.8	1	0	95.0	76.5	211.2	*
10	8.8	1	0	94.6	76.5	211.2	*
10	8.8	1	0	86.2	76.5	211.2	*
10	8.8	1	0	85.8	76.5	211.2	*
10	12.1	1	0	343.2	145.5	402.2	*
10	12.1	1	0	158.2	145.5	402.2	*
10	12.1	1	0	172.4	145.5	402.2	*
10	12.1	1	0	257.8	145.5	402.2	*
10	12.1	1	0	260.0	145.5	402.2	*
10	15.3	1	0	385.2	244.4	676.3	*
10	15.3	1	0	355.2	244.4	676.3	*
10	15.3	1	0	308.0	244.4	676.3	*
10	15.3	1	0	350.4	244.4	676.3	*
10	15.3	1	0	330.8	244.4	676.3	*
10	31.6	1	0	868.6	687.6	1,911.0	*
10	31.6	1	0	1,136.2	687.6	1,911.0	*
10	31.6	1	0	871.0	687.6	1,911.0	*
10	31.6	1	0	1,111.0	687.6	1,911.0	*
10	2.3	1	1	17.0	12.2	33.9	*
10	2.3	1	1	16.2	12.2	33.9	*
10	2.3	1	1	16.4	12.2	33.9	*
10	2.3	1	1	20.8	12.2	33.9	*
10	2.3	1	1	21.0	12.2	33.9	*

Table 1 continued

Temperature (°C)	TSS (g/l)	Paper type	Funnel geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
10	5.6	1	1	61.0	29.3	80.8	*
10	5.6	1	1	65.8	29.3	80.8	*
10	5.6	1	1	62.8	29.3	80.8	*
10	5.6	1	1	73.4	29.3	80.8	*
10	5.6	1	1	73.2	29.3	80.8	*
10	5.6	1	1	63.0	29.3	80.8	*
10	5.6	1	1	64.2	29.3	80.8	*
10	5.6	1	1	63.8	29.3	80.8	*
10	5.6	1	1	63.0	29.3	80.8	*
10	5.6	1	1	67.0	29.3	80.8	*
10	8.8	1	1	115.4	60.1	166.0	*
10	8.8	1	1	67.4	60.1	166.0	*
10	8.8	1	1	87.2	60.1	166.0	*
10	8.8	1	1	78.6	60.1	166.0	*
10	8.8	1	1	89.2	60.1	166.0	*
10	12.1	1	1	208.2	114.4	316.2	*
10	12.1	1	1	208.8	114.4	316.2	*
10	12.1	1	1	198.4	114.4	316.2	*
10	12.1	1	1	278.0	114.4	316.2	*
10	12.1	1	1	275.2	114.4	316.2	*
10	15.3	1	1	270.0	192.1	531.6	*
10	15.3	1	1	278.0	192.1	531.6	*
10	15.3	1	1	280.0	192.1	531.6	*
10	15.3	1	1	254.4	192.1	531.6	*
10	31.6	1	1	729.6	540.5	1,502.1	*
10	31.6	1	1	625.0	540.5	1,502.1	*
10	31.6	1	1	904.0	540.5	1,502.1	*
10	31.6	1	1	663.0	540.5	1,502.1	*
10	31.6	1	1	745.4	540.5	1,502.1	*
10	2.3	0	0	20.2	11.6	32.0	*
10	2.3	0	0	13.2	11.6	32.0	*
10	2.3	0	0	12.8	11.6	32.0	*
10	5.6	0	0	59.6	27.6	76.4	*
10	5.6	0	0	83.8	27.6	76.4	
10	5.6	0	0	52.0	27.6	76.4	*
10	5.6	0	0	48.2	27.6	76.4	*
10	5.6	0	0	47.2	27.6	76.4	*
10	5.6	0	0	45.8	27.6	76.4	*
10	5.6	0	0	55.8	27.6	76.4	*
10	5.6	0	0	40.8	27.6	76.4	*
10	5.6	0	0	45.0	27.6	76.4	*
10	5.6	0	0	52.4	27.6	76.4	*
10	8.8	0	0	66.4	56.8	156.9	*
10	8.8	0	0	93.0	56.8	156.9	*
10	8.8	0	0	95.2	56.8	156.9	*

Table 1 continued

Temperature (^oC)	TSS (g/l)	Paper type	Funnel geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
10	12.1	0	0	133.6	108.0	298.8	*
10	12.1	0	0	136.0	108.0	298.8	*
10	12.1	0	0	129.8	108.0	298.8	*
10	15.3	0	0	302.4	181.4	502.4	*
10	15.3	0	0	409.4	181.4	502.4	*
10	15.3	0	0	347.4	181.4	502.4	*
10	31.6	0	0	663.4	510.3	1,419.8	*
10	31.6	0	0	827.4	510.3	1,419.8	*
10	31.6	0	0	792.4	510.3	1,419.8	*
10	2.3	0	1	21.8	9.1	25.2	*
10	2.3	0	1	18.8	9.1	25.2	*
10	2.3	0	1	16.2	9.1	25.2	*
10	5.6	0	1	32.9	21.7	60.0	*
10	5.6	0	1	28.9	21.7	60.0	*
10	5.6	0	1	32.1	21.7	60.0	*
10	5.6	0	1	31.2	21.7	60.0	*
10	5.6	0	1	33.1	21.7	60.0	*
10	5.6	0	1	35.8	21.7	60.0	*
10	5.6	0	1	26.2	21.7	60.0	*
10	5.6	0	1	37.2	21.7	60.0	*
10	5.6	0	1	25.0	21.7	60.0	*
10	5.6	0	1	30.2	21.7	60.0	*
10	8.8	0	1	61.6	44.6	123.3	*
10	8.8	0	1	62.4	44.6	123.3	*
10	8.8	0	1	66.2	44.6	123.3	*
10	12.1	0	1	136.4	84.9	234.9	*
10	12.1	0	1	114.0	84.9	234.9	*
10	12.1	0	1	117.8	84.9	234.9	*
10	15.3	0	1	301.6	142.6	395.0	*
10	15.3	0	1	270.2	142.6	395.0	*
10	15.3	0	1	278.4	142.6	395.0	*
10	31.6	0	1	828.6	401.2	1,116.0	*
10	31.6	0	1	791.8	401.2	1,116.0	*
10	31.6	0	1	811.8	401.2	1,116.0	*
15	2.3	1	0	16.8	11.8	32.6	*
15	2.3	1	0	17.8	11.8	32.6	*
15	2.3	1	0	17.2	11.8	32.6	*
15	2.3	1	0	15.2	11.8	32.6	*
15	2.3	1	0	17.4	11.8	32.6	*
15	5.6	1	0	53.6	28.2	77.7	*
15	5.6	1	0	67.6	28.2	77.7	*
15	5.6	1	0	57.4	28.2	77.7	*
15	5.6	1	0	58.2	28.2	77.7	*
15	5.6	1	0	76.2	28.2	77.7	*
15	5.6	1	0	45.8	28.2	77.7	*

Table 1 continued

Temperature (^oC)	TSS (g/l)	Paper type	Funnel geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
15	5.6	1	0	63.6	28.2	77.7	*
15	5.6	1	0	66.8	28.2	77.7	*
15	5.6	1	0	59.8	28.2	77.7	*
15	5.6	1	0	60.0	28.2	77.7	*
15	5.6	1	0	61.0	28.2	77.7	*
15	8.8	1	0	77.8	57.9	159.6	*
15	8.8	1	0	74.4	57.9	159.6	*
15	8.8	1	0	79.0	57.9	159.6	*
15	8.8	1	0	81.2	57.9	159.6	*
15	8.8	1	0	85.2	57.9	159.6	*
15	12.1	1	0	164.4	110.2	303.9	*
15	12.1	1	0	143.4	110.2	303.9	*
15	12.1	1	0	152.0	110.2	303.9	*
15	12.1	1	0	156.2	110.2	303.9	*
15	12.1	1	0	156.4	110.2	303.9	*
15	15.3	1	0	313.8	185.0	511.1	*
15	15.3	1	0	301.2	185.0	511.1	*
15	15.3	1	0	260.0	185.0	511.1	*
15	15.3	1	0	354.8	185.0	511.1	*
15	15.3	1	0	313.8	185.0	511.1	*
15	31.6	1	0	1,009.2	520.5	1,444.1	*
15	31.6	1	0	1,001.8	520.5	1,444.1	*
15	31.6	1	0	1,083.0	520.5	1,444.1	*
15	31.6	1	0	904.8	520.5	1,444.1	*
15	2.3	1	1	15.2	9.3	25.6	*
15	2.3	1	1	16.6	9.3	25.6	*
15	2.3	1	1	17.8	9.3	25.6	*
15	2.3	1	1	18.0	9.3	25.6	*
15	2.3	1	1	15.2	9.3	25.6	*
15	5.6	1	1	35.6	22.2	61.1	*
15	5.6	1	1	43.0	22.2	61.1	*
15	5.6	1	1	43.2	22.2	61.1	*
15	5.6	1	1	44.2	22.2	61.1	*
15	5.6	1	1	36.0	22.2	61.1	*
15	5.6	1	1	36.6	22.2	61.1	*
15	5.6	1	1	33.8	22.2	61.1	*
15	5.6	1	1	27.4	22.2	61.1	*
15	5.6	1	1	29.0	22.2	61.1	*
15	8.8	1	1	67.4	45.5	125.5	*
15	8.8	1	1	61.4	45.5	125.5	*
15	8.8	1	1	51.4	45.5	125.5	*
15	8.8	1	1	76.8	45.5	125.5	*
15	8.8	1	1	69.2	45.5	125.5	*
15	12.1	1	1	154.4	86.6	238.9	*
15	12.1	1	1	181.6	86.6	238.9	*

Table 1 continued

Temperature (°C)	TSS (g/l)	Paper type	Funnel geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
15	12.1	1	1	204.6	86.6	238.9	*
15	12.1	1	1	219.2	86.6	238.9	*
15	12.1	1	1	225.6	86.6	238.9	*
15	15.3	1	1	258.4	145.4	401.7	*
15	15.3	1	1	272.4	145.4	401.7	*
15	15.3	1	1	251.6	145.4	401.7	*
15	15.3	1	1	281.4	145.4	401.7	*
15	15.3	1	1	270.4	145.4	401.7	*
15	31.6	1	1	770.6	409.2	1,135.1	*
15	31.6	1	1	790.6	409.2	1,135.1	*
15	31.6	1	1	730.2	409.2	1,135.1	*
15	31.6	1	1	815.2	409.2	1,135.1	*
15	31.6	1	1	687.6	409.2	1,135.1	*
15	2.3	0	0	12.4	8.8	24.2	*
15	2.3	0	0	12.6	8.8	24.2	*
15	2.3	0	0	10.8	8.8	24.2	*
15	5.6	0	0	38.8	20.9	57.7	*
15	5.6	0	0	36.2	20.9	57.7	*
15	5.6	0	0	46.0	20.9	57.7	*
15	5.6	0	0	39.4	20.9	57.7	*
15	5.6	0	0	37.8	20.9	57.7	*
15	5.6	0	0	43.0	20.9	57.7	*
15	5.6	0	0	47.0	20.9	57.7	*
15	5.6	0	0	51.4	20.9	57.7	*
15	5.6	0	0	35.2	20.9	57.7	*
15	5.6	0	0	41.0	20.9	57.7	*
15	8.8	0	0	62.4	43.0	118.5	*
15	8.8	0	0	39.0	43.0	118.5	
15	8.8	0	0	68.4	43.0	118.5	*
15	12.1	0	0	96.2	81.8	225.8	*
15	12.1	0	0	171.0	81.8	225.8	*
15	12.1	0	0	104.0	81.8	225.8	*
15	15.3	0	0	192.0	137.3	379.7	*
15	15.3	0	0	202.6	137.3	379.7	*
15	15.3	0	0	179.0	137.3	379.7	*
15	31.6	0	0	954.6	386.3	1,072.9	*
15	31.6	0	0	739.8	386.3	1,072.9	*
15	31.6	0	0	758.6	386.3	1,072.9	*
15	2.3	0	1	14.6	6.9	19.0	*
15	2.3	0	1	12.4	6.9	19.0	*
15	2.3	0	1	11.8	6.9	19.0	*
15	5.6	0	1	36.4	16.4	45.4	*
15	5.6	0	1	23.8	16.4	45.4	*
15	5.6	0	1	32.6	16.4	45.4	*
15	5.6	0	1	24.0	16.4	45.4	*

Table 1 continued

Temperature (°C)	TSS (g/l)	Paper type	Funnel geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
15	5.6	0	1	34.0	16.4	45.4	*
15	5.6	0	1	28.4	16.4	45.4	*
15	5.6	0	1	30.6	16.4	45.4	*
15	5.6	0	1	28.0	16.4	45.4	*
15	5.6	0	1	34.4	16.4	45.4	*
15	5.6	0	1	36.2	16.4	45.4	*
15	8.8	0	1	39.2	33.8	93.2	*
15	8.8	0	1	46.4	33.8	93.2	*
15	8.8	0	1	42.6	33.8	93.2	*
15	12.1	0	1	99.8	64.3	177.5	*
15	12.1	0	1	82.2	64.3	177.5	*
15	12.1	0	1	99.4	64.3	177.5	*
15	15.3	0	1	145.4	108.0	298.5	*
15	15.3	0	1	154.0	108.0	298.5	*
15	15.3	0	1	148.2	108.0	298.5	*
15	31.6	0	1	647.4	303.7	843.3	*
15	31.6	0	1	696.6	303.7	843.3	*
15	31.6	0	1	781.8	303.7	843.3	*
20	2.3	1	0	17.0	12.5	34.6	*
20	2.3	1	0	16.6	12.5	34.6	*
20	2.3	1	0	16.8	12.5	34.6	*
20	2.3	1	0	13.0	12.5	34.6	*
20	2.3	1	0	16.0	12.5	34.6	*
20	5.6	1	0	71.4	29.9	82.5	*
20	5.6	1	0	41.8	29.9	82.5	*
20	5.6	1	0	42.6	29.9	82.5	*
20	5.6	1	0	63.6	29.9	82.5	*
20	5.6	1	0	35.6	29.9	82.5	*
20	5.6	1	0	48.6	29.9	82.5	*
20	5.6	1	0	46.0	29.9	82.5	*
20	5.6	1	0	44.4	29.9	82.5	*
20	5.6	1	0	43.8	29.9	82.5	*
20	5.6	1	0	54.6	29.9	82.5	*
20	5.6	1	0	49.2	29.9	82.5	*
20	8.8	1	0	89.4	61.5	169.5	*
20	8.8	1	0	90.6	61.5	169.5	*
20	8.8	1	0	68.8	61.5	169.5	*
20	8.8	1	0	63.4	61.5	169.5	*
20	8.8	1	0	58.2	61.5	169.5	
20	12.1	1	0	174.6	117.0	322.8	*
20	12.1	1	0	170.2	117.0	322.8	*
20	12.1	1	0	163.4	117.0	322.8	*
20	12.1	1	0	162.2	117.0	322.8	*
20	12.1	1	0	172.4	117.0	322.8	*
20	15.3	1	0	353.2	196.5	542.8	*

Table 1 continued

Temperature (^oC)	TSS (g/l)	Paper type	Funnel geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
20	15.3	1	0	367.4	196.5	542.8	*
20	15.3	1	0	385.2	196.5	542.8	*
20	15.3	1	0	318.6	196.5	542.8	*
20	15.3	1	0	371.2	196.5	542.8	*
20	31.6	1	0	894.2	552.8	1,533.7	*
20	31.6	1	0	879.2	552.8	1,533.7	*
20	31.6	1	0	969.4	552.8	1,533.7	*
20	31.6	1	0	931.0	552.8	1,533.7	*
20	31.6	1	0	854.4	552.8	1,533.7	*
20	2.3	1	1	23.2	9.8	27.2	*
20	2.3	1	1	21.4	9.8	27.2	*
20	2.3	1	1	22.2	9.8	27.2	*
20	2.3	1	1	23.8	9.8	27.2	*
20	2.3	1	1	24.4	9.8	27.2	*
20	5.6	1	1	40.0	23.5	64.9	*
20	5.6	1	1	40.8	23.5	64.9	*
20	5.6	1	1	32.2	23.5	64.9	*
20	5.6	1	1	52.4	23.5	64.9	*
20	5.6	1	1	46.8	23.5	64.9	*
20	5.6	1	1	51.8	23.5	64.9	*
20	5.6	1	1	44.0	23.5	64.9	*
20	5.6	1	1	38.2	23.5	64.9	*
20	5.6	1	1	45.0	23.5	64.9	*
20	5.6	1	1	46.2	23.5	64.9	*
20	8.8	1	1	48.8	48.3	133.2	*
20	8.8	1	1	71.2	48.3	133.2	*
20	8.8	1	1	52.6	48.3	133.2	*
20	8.8	1	1	61.2	48.3	133.2	*
20	8.8	1	1	52.0	48.3	133.2	*
20	12.1	1	1	111.0	92.0	253.8	*
20	12.1	1	1	129.6	92.0	253.8	*
20	12.1	1	1	87.2	92.0	253.8	
20	12.1	1	1	104.6	92.0	253.8	*
20	12.1	1	1	78.0	92.0	253.8	
20	15.3	1	1	256.8	154.5	426.7	*
20	15.3	1	1	242.0	154.5	426.7	*
20	15.3	1	1	215.4	154.5	426.7	*
20	15.3	1	1	203.8	154.5	426.7	*
20	15.3	1	1	224.6	154.5	426.7	*
20	31.6	1	1	740.8	434.6	1,205.6	*
20	31.6	1	1	771.8	434.6	1,205.6	*
20	31.6	1	1	667.4	434.6	1,205.6	*
20	31.6	1	1	714.8	434.6	1,205.6	*
20	31.6	1	1	703.4	434.6	1,205.6	*
20	2.3	0	0	10.4	9.3	25.7	*

Table 1 continued

Temperature (°C)	TSS (g/l)	Paper type	Funnel geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
20	2.3	0	0	12.2	9.3	25.7	*
20	2.3	0	0	13.6	9.3	25.7	*
20	5.6	0	0	54.2	22.2	61.3	*
20	5.6	0	0	44.6	22.2	61.3	*
20	5.6	0	0	41.2	22.2	61.3	*
20	5.6	0	0	41.0	22.2	61.3	*
20	5.6	0	0	44.2	22.2	61.3	*
20	5.6	0	0	36.0	22.2	61.3	*
20	5.6	0	0	49.0	22.2	61.3	*
20	5.6	0	0	52.4	22.2	61.3	*
20	5.6	0	0	48.8	22.2	61.3	*
20	5.6	0	0	45.8	22.2	61.3	*
20	8.8	0	0	40.8	45.6	125.9	
20	8.8	0	0	47.8	45.6	125.9	*
20	8.8	0	0	33.0	45.6	125.9	
20	12.1	0	0	157.6	86.8	239.8	*
20	12.1	0	0	239.8	86.8	239.8	*
20	12.1	0	0	231.2	86.8	239.8	*
20	15.3	0	0	217.4	145.9	403.3	*
20	15.3	0	0	191.0	145.9	403.3	*
20	15.3	0	0	314.2	145.9	403.3	*
20	31.6	0	0	953.0	410.3	1,139.5	*
20	31.6	0	0	849.8	410.3	1,139.5	*
20	31.6	0	0	1,001.2	410.3	1,139.5	*
20	2.3	0	1	16.0	7.3	20.2	*
20	2.3	0	1	11.4	7.3	20.2	*
20	2.3	0	1	10.6	7.3	20.2	*
20	5.6	0	1	41.6	17.5	48.2	*
20	5.6	0	1	28.6	17.5	48.2	*
20	5.6	0	1	28.4	17.5	48.2	*
20	5.6	0	1	31.0	17.5	48.2	*
20	5.6	0	1	26.6	17.5	48.2	*
20	5.6	0	1	32.4	17.5	48.2	*
20	5.6	0	1	36.0	17.5	48.2	*
20	5.6	0	1	32.6	17.5	48.2	*
20	5.6	0	1	30.2	17.5	48.2	*
20	5.6	0	1	28.2	17.5	48.2	*
20	8.8	0	1	30.4	35.9	99.0	
20	8.8	0	1	33.4	35.9	99.0	
20	8.8	0	1	27.2	35.9	99.0	
20	12.1	0	1	145.0	68.3	188.5	*
20	12.1	0	1	160.6	68.3	188.5	*
20	12.1	0	1	170.4	68.3	188.5	*
20	15.3	0	1	164.8	114.7	317.0	*
20	15.3	0	1	243.0	114.7	317.0	*

Table 1 continued

Temperature (°C)	TSS (g/l)	Paper type	Funnel geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
20	15.3	0	1	144.4	114.7	317.0	*
20	31.6	0	1	798.0	322.6	895.7	*
20	31.6	0	1	734.4	322.6	895.7	*
20	31.6	0	1	706.6	322.6	895.7	*
25	2.3	1	0	25.2	18.7	51.7	*
25	2.3	1	0	27.2	18.7	51.7	*
25	2.3	1	0	25.6	18.7	51.7	*
25	2.3	1	0	22.2	18.7	51.7	*
25	2.3	1	0	26.2	18.7	51.7	*
25	5.6	1	0	57.4	44.6	123.2	*
25	5.6	1	0	87.3	44.6	123.2	*
25	5.6	1	0	77.2	44.6	123.2	*
25	5.6	1	0	54.4	44.6	123.2	*
25	5.6	1	0	67.6	44.6	123.2	*
25	5.6	1	0	75.7	44.6	123.2	*
25	5.6	1	0	70.2	44.6	123.2	*
25	5.6	1	0	72.7	44.6	123.2	*
25	5.6	1	0	68.3	44.6	123.2	*
25	5.6	1	0	57.6	44.6	123.2	*
25	8.8	1	0	252.2	91.6	253.0	*
25	8.8	1	0	266.4	91.6	253.0	
25	8.8	1	0	300.8	91.6	253.0	
25	8.8	1	0	294.6	91.6	253.0	
25	8.8	1	0	295.0	91.6	253.0	
25	12.1	1	0	462.4	174.4	481.9	*
25	12.1	1	0	385.4	174.4	481.9	*
25	12.1	1	0	452.6	174.4	481.9	*
25	12.1	1	0	463.6	174.4	481.9	*
25	12.1	1	0	465.8	174.4	481.9	*
25	15.3	1	0	411.0	292.9	810.3	*
25	15.3	1	0	391.2	292.9	810.3	*
25	15.3	1	0	421.2	292.9	810.3	*
25	15.3	1	0	483.2	292.9	810.3	*
25	15.3	1	0	496.8	292.9	810.3	*
25	31.6	1	0	1,095.4	823.9	2,289.5	*
25	31.6	1	0	997.4	823.9	2,289.5	*
25	31.6	1	0	1,289.4	823.9	2,289.5	*
25	31.6	1	0	1,191.6	823.9	2,289.5	*
25	31.6	1	0	1,154.8	823.9	2,289.5	*
25	2.3	1	1	25.2	14.7	40.6	*
25	2.3	1	1	21.6	14.7	40.6	*
25	2.3	1	1	25.6	14.7	40.6	*
25	2.3	1	1	22.4	14.7	40.6	*
25	2.3	1	1	25.2	14.7	40.6	*
25	5.6	1	1	44.2	35.1	96.8	*

Table 1 continued

Temperature (°C)	TSS (g/l)	Paper type	Funnel geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
25	5.6	1	1	47.8	35.1	96.8	*
25	5.6	1	1	47.2	35.1	96.8	*
25	5.6	1	1	42.1	35.1	96.8	*
25	5.6	1	1	44.8	35.1	96.8	*
25	5.6	1	1	51.1	35.1	96.8	*
25	5.6	1	1	53.8	35.1	96.8	*
25	5.6	1	1	59.1	35.1	96.8	*
25	5.6	1	1	49.2	35.1	96.8	*
25	5.6	1	1	46.1	35.1	96.8	*
25	8.8	1	1	110.4	72.0	198.9	*
25	8.8	1	1	133.0	72.0	198.9	*
25	8.8	1	1	139.4	72.0	198.9	*
25	8.8	1	1	138.8	72.0	198.9	*
25	8.8	1	1	165.8	72.0	198.9	*
25	12.1	1	1	352.8	137.1	378.8	*
25	12.1	1	1	367.6	137.1	378.8	*
25	12.1	1	1	383.2	137.1	378.8	
25	12.1	1	1	392.6	137.1	378.8	
25	12.1	1	1	387.2	137.1	378.8	
25	15.3	1	1	424.8	230.2	636.9	*
25	15.3	1	1	430.8	230.2	636.9	*
25	15.3	1	1	444.8	230.2	636.9	*
25	15.3	1	1	320.0	230.2	636.9	*
25	15.3	1	1	454.6	230.2	636.9	*
25	31.6	1	1	886.0	647.7	1,799.6	*
25	31.6	1	1	715.8	647.7	1,799.6	*
25	31.6	1	1	963.6	647.7	1,799.6	*
25	31.6	1	1	1,002.2	647.7	1,799.6	*
25	31.6	1	1	1,011.6	647.7	1,799.6	*
25	2.3	0	0	17.0	13.8	38.4	*
25	2.3	0	0	16.4	13.8	38.4	*
25	2.3	0	0	15.8	13.8	38.4	*
25	5.6	0	0	62.1	33.1	91.5	*
25	5.6	0	0	51.9	33.1	91.5	*
25	5.6	0	0	59.1	33.1	91.5	*
25	5.6	0	0	71.2	33.1	91.5	*
25	5.6	0	0	77.6	33.1	91.5	*
25	5.6	0	0	85.4	33.1	91.5	*
25	5.6	0	0	57.3	33.1	91.5	*
25	5.6	0	0	58.8	33.1	91.5	*
25	5.6	0	0	68.3	33.1	91.5	*
25	5.6	0	0	68.9	33.1	91.5	*
25	8.8	0	0	179.8	68.0	188.0	*
25	8.8	0	0	122.8	68.0	188.0	*
25	8.8	0	0	173.2	68.0	188.0	*

Table 1 continued

Temperature (^oC)	TSS (g/l)	Paper type	Funnel geometry	Observed CST estimates used to calibrate the model (s)	Lower limit of 95% prediction interval for CST (s)	Upper limit of 95% prediction interval for CST (s)	Observed CST is within the 95% prediction intervals of the model
25	12.1	0	0	182.4	129.4	358.0	*
25	12.1	0	0	237.0	129.4	358.0	*
25	12.1	0	0	269.0	129.4	358.0	*
25	15.3	0	0	375.6	217.4	602.0	*
25	15.3	0	0	344.0	217.4	602.0	*
25	15.3	0	0	335.6	217.4	602.0	*
25	31.6	0	0	793.6	611.5	1,701.0	*
25	31.6	0	0	829.0	611.5	1,701.0	*
25	31.6	0	0	797.2	611.5	1,701.0	*
25	2.3	0	1	15.6	10.9	30.2	*
25	2.3	0	1	16.4	10.9	30.2	*
25	2.3	0	1	14.4	10.9	30.2	*
25	5.6	0	1	39.9	26.0	71.9	*
25	5.6	0	1	36.9	26.0	71.9	*
25	5.6	0	1	40.2	26.0	71.9	*
25	5.6	0	1	27.9	26.0	71.9	*
25	5.6	0	1	31.2	26.0	71.9	*
25	5.6	0	1	45.0	26.0	71.9	*
25	5.6	0	1	40.5	26.0	71.9	*
25	5.6	0	1	36.3	26.0	71.9	*
25	5.6	0	1	25.5	26.0	71.9	
25	5.6	0	1	40.8	26.0	71.9	*
25	8.8	0	1	116.4	53.5	147.8	*
25	8.8	0	1	91.2	53.5	147.8	*
25	8.8	0	1	91.0	53.5	147.8	*
25	12.1	0	1	185.6	101.7	281.4	*
25	12.1	0	1	183.6	101.7	281.4	*
25	12.1	0	1	134.4	101.7	281.4	*
25	15.3	0	1	291.8	170.9	473.2	*
25	15.3	0	1	263.4	170.9	473.2	*
25	15.3	0	1	321.2	170.9	473.2	*
25	31.6	0	1	717.6	480.7	1,337.0	*
25	31.6	0	1	665.0	480.7	1,337.0	*
25	31.6	0	1	726.2	480.7	1,337.0	*

* The observed CST is within the 95% prediction intervals of the model; Paper type: 0 = Fisher 200 chr; 1 = Whatman 17 chr; Funnel geometry: 0 = circular; 1 = rectangular